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Cooperative Programme on Mechanical Testing of Refractory Metals

by

D. Coutsouradis

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1968

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NORTH ATLANTIC TREATY ORGANIZATION

ADVISORY GROUP FOR AEROSPACE RESEARCH AND DEVELOPMENT

(ORGANISATION DU TRAITE DE l'ATLANTIQUE NORD)

STRUCTURES AND MATERIALS PANEL

6 COOPERATIVE PROGRAMME ON MECHANICAL TESTING OF REFRACTORY METALS,

by

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FOREWORD

The Structures and Materials Panel of the NATO Advisory Group for Aerospace Research and Development (AGARD) is comprised of scientists, engineers and technical administrators, from industry, government and universities throughout NATO, concerned with advancing the status of aerospace research and development and with developing technical means and data for optimizing the vehicles and equipment of interest to NATO. The Panel, therefore, provides a discussion forum, a mechanism for exchanging information, and a means for establishing and conducting cooperative studies and laboratory programs in selected technical areas.

Clearly, one of the technical areas of prime concern to this Panel is the behavior of metals at elevated temperatures, particularly in critical applications such as propulsion (powerplants) and structures subject to high speed flight or adjacent to heat-generating devices. In planning the early stages of cooperative programs to study and determine the properties of metals at elevated temperatures, and in considering the exchange of available, high temperature data, it soon became apparent that the scatter which was evident in high temperature measurements must first be resolved, either in terms of material variability or differences in the details of testing techniques or both.

It was decided to first examine testing techniques using a "standard" and hopefully uniform material. Fortunately, it was recognized from the beginning that the examination in depth of any test would be both time-consuming and expensive. For a beginning, therefore, a "simple" test - the tensile test - was selected for a "round-robin" study in laboratories (a total of eleven) in a number of NATO countries; later some additional testing was added. The original familiarization work was done on tungsten, niobium, tantalum and molybdenum. After this preliminary evaluation of techniques, the more rigorous high-temperature testing was done on a molybdenum alloy. In order to assist in analyzing the scatter of results, tests were performed on a "standard" steel at room temperature, to eliminate high temperature variables.

This report contains the description, results and analysis of this extensive interlaboratory cooperative program. It is hoped that the results, conclusions and suggestions for improvements in testing specifications will serve as a useful contribution to more effective exchange of data, on a more reliable basis.

The Panel is indebted to the countries who contributed the materials for this program - the United Kingdom, France and the United States, to the many laboratories who freely contributed their time and talent, to Mr D.K.Faurschou for his statistical evaluation, and to Dr D.Coutsouradis, the coordinator of this project and the author of this report, who so conscientiously and so capably and patiently guided this intricate project to its successful conclusion.

N.E.Promisel
Chairman
AGARD Structures & Materials Panel

Washington, D.C.
January 31, 1969

the REPORT DEALS WITH:

SUMMARY

The interlaboratory cooperative program, in which 11 laboratories from different NATO countries have participated, dealt with:

- the determination of room temperature tensile properties of Vascojet 90 (15CDV6) steel sheet, for conventional testing techniques ;
- the same, under high temperature testing conditions ;
- the determination of the recrystallization temperature of TZM molybdenum alloy sheet ; and
- the determination of the tensile properties of TZM molybdenum alloy sheet at RT, 1050°, 1450° and 1800°C.

The objective of the program was to generate information on the adequacy of test techniques and specifications and also on the properties of the materials tested. The report reproduces data on the variability of test results within and between laboratories. For most of the tests performed the interlaboratory variability was significantly larger than the intralaboratory one.

The adequacy of test techniques and specifications is discussed in terms of the available results. Suggestions are made on the modifications of the original specifications.

RESUME

Le programme coopératif d'essais, auquel ont participé 11 laboratoires de différents pays de l'OTAN, a porté sur:

- la détermination, à la température ambiante, des propriétés de traction de tôles en acier 15CDV6 (Vascojet 90), par des techniques conventionnelles
- la même détermination par les techniques utilisées pour les essais à haute température
- la détermination de la température de recristallisation de tôles en alliage de molybdène TZM
- la détermination des propriétés de traction de tôles en alliage de molybdène TZM à la température ambiante, 1050°C, 1450°C, et 1800°C.

Le programme visait à évaluer les possibilités d'amélioration des techniques expérimentales et des spécifications d'essais ainsi qu'à obtenir des renseignements sur les propriétés des matériaux utilisés. Le rapport présente des données permettant d'évaluer la dispersion des résultats à l'intérieur même des laboratoires ou entre ceux et ci. Les données obtenues permettent de tirer des conclusions quant aux conditions des essais et de suggérer quelques modifications aux spécifications initiales utilisées.

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The implementation of the cooperative programme has been possible through the continuing encouragement and support of the members of the Structures and Materials Panel of AGARD and the active cooperation of the Panel Executive. The contribution of the delegates of US., UK and France who made available the experimental materials is especially acknowledged. The cooperativeness of the participating laboratories has been essential in the fulfilment of the collaborative programme.

The statistical evaluation of the results profited much from fruitful discussions with Mr D.K.FAURSCHOU.

COOPERATIVE PROGRAMME ON MECHANICAL TESTING OF REFRACTORY METALS

D.Coutsouradis

1. INTRODUCTION

The present report reproduces the results obtained within the frame of the Materials Group Cooperative programme on mechanical testing of refractory metals. The purposes of the programme were:

(a) to verify the adequacy of the specifications used for the tests.

(b) to determine the relative precision and accuracy of the test methods.

(c) to provide data for the eventual improvement of the specifications or the test methods.

In the early stages of the programme various methods used for the evaluation of refractory metals were considered. Taking into account that the materials and the tests are expensive only a limited number of tests were selected; namely: tensile tests at various temperatures and recrystallization temperature tests. Since one of the objectives of the programme was to establish the scatter of data within one laboratory, repetitive tests were requested from the various laboratories.

The materials considered in this program included a TZM molybdenum alloy sheet and a Vascojet 90 steel sheet. Table I summarizes the type of tests carried out on the two materials. The testing on the Vascojet 90 steel sheet was introduced in order to put in evidence any systematic effect of the high temperature equipment on the tensile properties. Also, in the very early stages of the programme sheets of W, Nb, Ta and Mo were made available to the Panel by UK. These have been used by a number of the participating laboratories for the purpose of the preliminary evaluation of the specifications. The results obtained in this preliminary stage have not been systematically reported and will not be reproduced here.

The documents used, which include the definition of the materials and the specifications, are reproduced in Appendix A and B respectively for the TZM material and the Vascojet 90 steel.

A total of eleven laboratories participated in this programme. They were subdivided within various NATO countries as shown in Table 2.

In the presentation of the results the data reported are referred to the originating laboratory in an anonymous way.

The results are presented for uniformity in metric units. The tensile strength data originally given in psi were converted to kg/mm^2 by multiplying by the factor 7.03×10^{-4}. The discussion of the data was performed on the basis of direct comparisons or of simple statistical evaluations The conclusions deal with the material properties, the adequacy of experimental methods and suggestions for the improvement of the original specifications.

2. MATERIALS

2.1 TZM Molybdenum Alloy Sheet

The TZM sheet material was made available to the Materials Group through the courtesy of USA. It was produced by the Universal Cyclops Steel Corporation.

Data on the fabrication, composition and presentation of the material may be found in Appendix A. Furthermore, Figure 1 reproduces the scheme of cutting and indicates the pieces tested in the different laboratories. The original sheets were cut by the producer. They were numbered 1 through 18. Each laboratory used one piece of 600 x 200 mm^2 except for laboratories II, IV and XI which used two sheets. The remaining 4 sheets have been used for additional testing in various places.

The variability in the thickness of the sheets was determined by some laboratories. For example laboratory VIII (sheet No. 10) determined a thickness of 1.45 mm ±0.08 mm, the extreme values being 1.37 and 1.53 mm. Laboratory X determined a mean thickness of 1.44 mm with extreme values of 1.375 and 1.500. The general aspect of the individual sheets was generally considered as satisfactory. However, traces of grinding were reported to be visible on some of them as delivered sheets. Furthermore, some of the individual sheets exhibited a slight bending.

The tests were carried out on specimens machined in the transverse direction of the original sheet. However laboratory VI used longitudinal specimens and laboratory XI used both.

Table 3 indicates the type of testing performed in each laboratory.

Sheets originating from the same heat and processed in a similar way as those used in this programme, were submitted to various evaluation tests by the producer. The producer's report is given in Appendix C. For convenience Table IV reproduces the transverse and longitudinal tensile data at RT of sheets 57 and 58 measured at two ends.

For each sheet the values obtained for end A and end B do not show appreciable difference except for the elongation values in the longitudinal direction. For both sheets it appears that the transverse tensile properties are higher and the elongation lower than the longitudinal properties. The difference observed between the sheets might not be significant.

Recently the Southern Research Institute published the results of a complete evaluation of TZM sheet material processed by the same Company and in the same way as the sheets used in this program[1]. The latter work confirms that the transverse UTS and YS properties of the TZM sheet material are higher than the longitudinal ones. The same work showed that the transverse elongation at RT of 0.06 in. thick sheet is appreciably lower than the longitudinal one. Data from this work were used to evaluate the within sheets, between sheets and between heats variability, for 0.060 in. thick sheets. The results of this evaluation are reproduced in Appendix C. For this type of material the within sheet or between sheets variability is only slightly significant.

2.2 Vascojet 90 Steel Sheet

The steel material was made available to the Materials Group by Sud Aviation through the courtesy of France.

The nominal composition of the Vascojet 90 steel (AFNOR-15CDV6) is as follows:

C: 0.10, 0.16 - Mn: 0.80, 1.00 - Si: 0.2 max - P 0.03 max - S: 0.03 max - Cr 1.25, 1.45 - Mo: 0.80 - 0.90 - V: 0.20, 0.30.

The actual composition of the sample was not made available. The sheet was delivered in the annealed condition. The original piece of about 1,500 × 500 × 1.5 mm^3 was cut into 500 by 150 mm pieces as shown in Appendix B. In Appendix B are reproduced also the mechanical evaluation properties as given by Sud Aviation and also the sampling method that prevailed for all laboratories.

The distribution of the pieces to the different laboratories is given in Figure 2.

The homogeneity of the original sheet may be assessed by the data given in Table 5, calculated from the measurements carried out by Sud Aviation.

3. TEST SPECIMENS

The test pieces used were generally in agreement with the recommendations, as to the gauge length used. In Figure 3a and 3b the configuration of some typical specimens is shown. The specimens used by laboratory VII have also been used by laboratories I, II, III, V, VI and XI with slight modifications to allow for the adaptation of an extensometer or to accomodate in the particular furnace considered. The specimens used by laboratory X were especially studied in order to minimize stress concentrations and to allow for an extensometer. The specimens used by laboratory IV had in fact a gauge length of two inches. Finally, specimen IX was designed for better alignment and for the measurement of the elongation at the ends of the test pieces.

4. EXPERIMENTAL TECHNIQUES

Table 6 summarizes the techniques used by the different laboratories for the room temperature tests. Table 7 summarizes the techniques used for the elevated temperature tests, For a more detailed survey, Appendix E reproduces in extenso the description of the experimental techniques as given in the individual laboratory reports.

The tensile equipment was generally of the mechanically driven type either commercially available or home-made. In some cases also hydraulic machines were used.

Loading was controlled either through the cross head speed or through the strain rate. Similarly, strain was measured either through the cross head movement or through an extensometer.

For the tests at elevated temperature, generally a resistor type furnace was used. However in one case self-resistance heating was employed and in another an electron beam furnace. In all cases the environment was vacuum.

For the temperature measurements at 1050°C chromel-alumel or Pt-Pt Rh thermocouples were used. At 1450°C the thermocouples used were of the Pt-Pt Rh type, or W-W Re type. At this temperature also optical pyrometers were used. At 1800°C, temperature was measured by means of optical pyrometers or W-W Re, W-Mo and W-Ir thermocouples.

5. RESULTS ON THE VASCOJET 90 STEEL SHEET

5.1 Conventional Room Temperature Tests

Tables 8, 9 and 10 reproduce the test data for respectively the Ultimate Tensile Strength, the 0.2% Yield Strength and Elongation for the Vascojet 90 steel sheet material as measured by conventional methods. The Tables give also the range, the variance and the standard deviation for each group of measures. Table 11 refers to the values of elastic modulus as reported by 3 laboratories.

5.2 Room Temperature Tests under High Temperature Test Conditions

Tables 12, 13 and 14 reproduce the values of tensile test data carried out under the same conditions as the high temperature tests. Table 15 gives the corresponding values for the elastic modulus. Comparing Tables 11 and 15, it is visible that only laboratories X and XI determined the elastic modulus of Vascojet steel for both testing conditions. Laboratories II and IV carried out the determination of modulus for one condition, respectively by conventional testing and under high-temperature testing conditions.

6. RESULTS OF THE TZM MOLYBDENUM ALLOY STEEL

6.1 Recrystallization Temperature

Table 16 gives the data relevant to the determination of the recrystallization temperature. For comparison the Tables gives also the temperature determined by the producer on sheets 57 and 58 of heat No.11.

6.2 Tensile Tests at Room Temperature

The results obtained at room temperature are shown in Tables 17, 18 and 19. Laboratory II reported results of tests carried out on two sheets with, respectively, two different tensile equipments. The results reported by laboratory VI were determined in the longitudinal direction. Laboratory XI reported both longitudinal and transverse properties determined respectively on two different sheets. All other results are, as stated previously, transverse.

Five laboratories reported also the elastic modulus. The results are reproduced in Table 20. Laboratory XI determined the modulus in the transverse as well as in the longitudinal direction.

6.3 Tensile Tests at 1050°C

Tables 21, 22 and 23 reproduce the tensile test data at 1050°C for the TZM molybdenum alloy sheet. As previosuly mentioned, laboratory VI determined the properties in the longitudinal direction. Laboratory XI determined the properties in the transverse direction for sheet No.7 and in the longitudinal one for sheet No.8. The latter determinations were carried out with an electron beam furnace and an optical extensometer. Under these conditions, the results of elastic modulus and yield strength were not considered satisfactory and they have not been reported. Table 24 reproduces the results of the elastic modulus.

6.4 Tensile Tests at 1450°C

Tables 25 to 28 reproduce the data dealing with the determination of tensile properties of TZM sheet at 1450°C. The same remarks prevail here as for the tests at 1050°C.

6.5 Tensile Tests at 1800°C

The results obtained at 1800°C are reproduced in Tables 29 to 32. Concerning the laboratories VI and XI, the same remarks are valid here as for the tests at 1050°C.

7. DISCUSSION OF THE RESULTS

7.1 Introduction

For the presentation of the results we have followed the recommendations of ASTM's "Manual for Conducting an Interlaboratory Study of a Test Method"[2]. According to this

manual a presentation based on control charts provides a good means for a preliminary evaluation of the results. The advantage of such a procedure is that it requires only simple calculations and little knowledge of elaborate statistical analysis. It serves thus only as a guide for the interpretation of the results, since the evaluation is only qualitative. A more quantitative approach based on analysis of variance techniques is given in Appendix F.

For the establishment of the Control charts the method proposed by DIXON and MASSEY[3] was used. Other references suggest slightly different methods.

The upper and lower limits of the control charts for ranges were calculated by multiplying the average values of ranges by, respectively, 1.76 and 0.441. These values correspond to 10 tests in each group and to a probability of 0.99. Although in some cases the number of tests in each group was less than 10, this latter value reduces the width of the probable domain and thus the criteria of evaluation are more severe. When the range of a series of measures carried out in one laboratory is situated within the limits defined above, it may be concluded that for the laboratory considered the factors affecting tests results are well under control. It is clear that the lower limit for range has little physical meaning. Ranges below this limit indicates only exceptionally good reproducibility.

If $\bar{X}$ is the overall average of several groups of measures and $\bar{R}$ the average value of the ranges, the upper and lower limits in the control chart for averages are given by: $\bar{X} \pm 0.31 \times \bar{R}$. Here again the factor 0.31 corresponds to 10 tests per group and to a probability of 0.99.

If the averages corresponding to different laboratories lie within the limits defined above, it may be concluded that within the laboratories considered the factors affecting test results are controlled in a similar way, provided that the material subjected to the tests is homogeneous. On the contrary, if the averages of some laboratories lie outside the control limits it may be considered that either the material is not homogeneous or that the factors affecting test results are not controlled in the same way.

Thus the control chart for ranges provides information on the precision* of the tests in each laboratory whereas the control chart for averages gives information of the precision of results among laboratories.

It should be once more emphasized here that the control charts are used only to assess whether the interlaboratory variability is of the same order of magnitude as the intra-laboratory one.

7.2 Vascojet 90 Steel Sheet

Tables 33 and 34 summarize the average values and the ranges for the tensile properties of Vascojet 90 steel sheet determined under, respectively, conventional and high temperature conditions.

Figure 4 reproduces the control charts for the tests carried out under conventional conditions. For UTS, YS and elongation the precision in each laboratory is satisfactory as shown by the control charts for ranges. Also, the variability among laboratories is satisfactory concerning the yield strength. It is less good for the UTS and elongation measurements. Laboratories I, VIII and XI obtained low values of UTS whereas the values reported by III and VI are rather high

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* The term "precision" is used here and in following sections as synonymous with reproducibility and not synonymous with accuracy. This terminology is in agreement with ASTM recommendations (ASTM E 177-61T).

The comparison of the mean values of yield strength (Table 9) to those given in Table I, Appendix B, shows, to some extent a systematic effect of the location of the sheets delivered to the different laboratories.

Examination of the test conditions as summarized in Table 6, do not allow one to trace the causes of these variabilities. For example, strain rate or cross head speed are not responsible for differences in values of UTS.

As to the elastic modulus, values given by 3 laboratories show differences which appear to be due only to random scatter.

Figure 5 gives the control charts for the tests carried out under high temperature conditions. The precision in each laboratory appears satisfactory except for laboratory XI which obtained slightly high ranges for UTS and YS. The variability within laboratories, as shown in the control charts for averages, appears appreciable. Comparing with Figure 4 it appears furthermore that the variability of 0.2% YS has increased.

It might be useful to compare the YS values obtained with the use of an extensometer with those obtained without an extensometer. Table 35 summarizes the relevant data as obtained in this programme. So far as the Vascojet 90 steel is considered, it appears that the values obtained with extensometer are slightly higher than those obtained by means of the cross head movement. It remains, however, to demonstrate that this difference is significant.

The examination of the overall data by the analysis of variance techniques, shows that neither the intralaboratory variability nor the interlaboratory one have been significantly affected for the tests carried out under elevated temperature conditions.

7.3 Tensile Tests on TZM at RT

The results on the RT tensile properties of the TZM sheet may be subdivided into two groups: the first which comprises the large majority of results refers to the transverse properties of the original sheet; the second refers to the longitudinal properties.

From the data given in Tables 17, 18 and 19, the averages and ranges are reproduced in Table 36. The overall averages and the average ranges have been also calculated and indicated in Table 36. Table 37 summarizes similar data for the measurements in the longitudinal direction.

The control charts relative to these data are shown in Figure 6.

The control chart for ranges indicates that the UTS is well controlled within each of all laboratories except for laboratory VIII. The control chart for averages shows that the dispersion among laboratories is higher than that which could be expected on the basis of the average scatter encountered within each laboratory. In particular, Laboratories I, III and XI obtained UTS values below the lower control limit whereas laboratories IV, VIII, IX and X obtained values higher than the upper control limit.

Except for laboratories IIb and VIII the control for the 0.2% YS is adequate. Here again laboratories I, III and XI obtained low values, while laboratories IV and VIII obtained high values. Laboratory IV has used 2 in. gauge length specimens whereas laboratory VIII used a cross head speed of 0.5 mm/min. throughout the test. Laboratory I on the contrary used the lowest cross head speed of 0.1 mm/min

The precision of the elongation values is good within each laboratory as well as among laboratories. The low values obtained by laboratories III and IV should not in fact be taken into consideration, because in the first case only two tests were carried out and [illegible] the second, the elongation values refer to a two inch gauge length. For TZM sheet at [illegible] a large part of the elongation values originates from local necking

The scatter of the average values for modulus is rather small. The scatter for laboratory II(a) was reported to be due to an imperfect and non-reproducible position of the extensometer on the specimen.

The longitudinal properties obtained by laboratories XI(a) and VI have not been considered in the calculation of control chart data. They have been plotted in the figures and marked by full circles. The control within each of these laboratories is similar to that in the other laboratories. However, UTS and more particularly 0.2 YS are in the high range whereas elongation values appear to be in the lower range.

It is difficult to draw a conclusion concerning the modulus. In fact laboratory XI(a) obtained as could be expected an average value within the control limits, Laboratory VI, obtained on the contrary a much lower value.

7.4 Tensile Tests on TZM at 1050°C

The control charts for averages and ranges drawn on the basis of data of Tables 38 and 39 are reproduced in Figure 7. The control for UTS, YS and elongation within each for the participating laboratories is adequate except in some rare cases: laboratory XI for UTS and laboratory II for YS. UTS is low for laboratory IV and high for laboratory IX. YS is low for laboratories, I, II and IX and high for laboratories IV, VIII and X. The control of YS among laboratories is rather poor. Elongation values are slightly high for laboratories I and II and low for laboratory IV. The latter observation is easily understood since laboratory IV used self-resistance heating which, as is well known, affects elongation values. The precision on Modulus (Table 38) appears satisfactory both concerning the tests within each laboratory or among laboratories.

In Figure 7 the data on longitudinal properties determined by laboratory XI(a) and III are plotted by full circles. Laboratory XI(a) has used in these tests an electron beam surface and an optical extensometer. In view of difficulties encountered with the latter equipment the YS values were not reported. UTA and YS values for laboratory VI appear well above the respective upper control limits, while elongation falls within the limits. The UTS value obtained by XI(a) appears low.

7.5 Tensile Tests on TZM Sheet at 1450°C

The data useful for comparison purposes are gathered in Tables 40 and 41 and the corresponding control charts are reproduced in Figure 8. The results of laboratory IV have not been included in the calculations because they are obviously too different from the other; UTS and YS are high whereas elongation is low. Laboratory IV has used self-resistance heating and also, a short time of heating (76 sec) and short holding time at temperature (3 min). From the data in Table 16, it appears that the test temperature of 1450°C is only about 100°C above the recrystallization temperature of the TZM alloy sheet. It is thus probable that for the short time at temperature used by laboratory IV only limited, if any, recrystallization has occurred. To assess for possible similar effects in the results of other laboratories the tensile test data are plotted in Figure 9 as a function of heating time plus time at temperature. Although the recrystallization behaviour of 1450°C played a role in the scatter of the results its effect was masked, to some extent, by that of other variables. There is a slight tendency for the UTS values to decrease when the holding at temperature increases above 45 min.

From the control charts in Figure 8 it is apparent that laboratories III and VII are in less good control for UTS and YS than the other laboratories. UTS is low for laboratory I and high for laboratory III. YS is low for laboratory III, VIII and IX. Elongation is low for laboratory III and high for laboratories IX, X and XI. The results of III compared to those of IV indicate that in the former the conditions of testing favoured an incomplete recrystallization. For the testing temperature considered here, the high values of

elongation obtained by laboratories IX, X and XI may well be due to an adequate temperature control along the gauge length.

The modulus values shown in Table 40 indicate a fairly low variability.

Longitudinal UTS values appear to be higher than the upper control limits. The longitudinal YS obtained by laboratory VI is within the control limits. Elongation in the longitudinal direction is low for laboratory XI(a). This low value may be due to unfavourable temperature gradient along the gauge length in view of the special heating device (electron beam) used.

7.6 Tensile Tests on TZM Sheet at 1800°C

From the results summarized in Tables 42 and 43 from the control charts in figure 10 it appears that the precision within each of the laboratories is well under control. The control among laboratories is, however, less good. Laboratory VIII obtained high values for UTS and YS probably because of the high cross head speed used. The high values of elongation obtained by laboratory X are attributed to a good control of the temperature along the gauge length of the specimen during elongation.

The longitudinal properties do not reveal any marked systematic effect. The low values of elongation obtained by laboratory XI(a), where an electron beam furnace was used, is due to inadequate control of the temperature gradient while the specimen elongates. The modulus values (Table 42) determined in two laboratories appear to be comparable.

7.7 Recrystallization Temperature

The data reproduced in Table 16 indicate that the values determined for the recrystallization temperature vary within the range of 1325°C to 1375°C. According to producer's data which are shown in Table 16, it is apparent that rather wide variations in recrystallization temperature can be encountered. Thus, a difference of more than 50°C exists between the two ends of sheet No.57. According to DOTSON[1] the recrystallization temperature of 0.060 in. thick TZM sheet similarly processed to that considered in this programme is of 1290°C or 1288°C. In the latter investigation, however, the within sheet or between sheets variability of recrystallization temperature was not investigated.

The variability among the laboratories in the recrystallization temperature values is rather large. It probably originates from physical or chemical inhomogeneities in the material. Recrystallization temperature is actually very sensitive to these factors.

7.8 Evaluation of Contamination During Testing

The specifications used requested, for the assessment of the contamination during testing at elevated temperature, the chemical analysis for oxygen, nitrogen, hydrogen, and carbon of chips comprising a cut of from 0.005 to 0.010 inch (0.127 to 0.250 mm) from the surface. It is well known that the chemical analysis in refractory metals of the above mentioned elements is a major problem particularly at low content levels. Thus only two of the participating laboratories provided data on the contamination of the TZM sheet after the elevated temperature tests.

Laboratory VIII performed the analysis of the four elements on massive specimens as well as on chips representing a cut of 0.15 mm from the surface. The analytical methods used were the following:

oxygen:	reducing melting under argon
nitrogen:	distillation of NH_3 and colorimetry with Nessler's reagent
hydrogen:	vacuum extraction at 1100°C
carbon:	combustion and coulometry.

The results of these determinations are reproduced in Table 44.

Examination of these results show that:

- there is no contamination by hydrogen
- the possible contamination by carbon is doubtful
- there is a slight surface contamination by nitrogen
- the contamination by oxygen is observable on the massive as well as on the chip samples.

Laboratory IX carried out the determination of carbon on massive specimens by combustion and coulometry. In the as delivered condition the carbon content was found to be of 250 ppm. After testing at RT, 1050°C, 1450°C or 1800°C the carbon content was found to be of 200 ppm. These results also indicate that no contamination by carbon is occurring during the high temperature testing of TZM sheet in vacuum.

7.9 Discussion of Test Techniques

In this section some aspects relevant to the test techniques used in the cooperative programme will be discussed. It may in fact be considered that the state of the art in the field of test-techniques plays a major role in the establishment of the specifications. Taking this consideration into account the discussion will be a function of the different parameters specified in the specifications.

7.9.1 Test Pieces

The 1 in. gauge-length specimens recommended in the specifications were adopted in order to save experimental material and also to reduce difficulties encountered in high-temperature testing. As mentioned in a previous section all laboratories but one (Lab.IV) used this type of specimen. Laboratory IX used tensile specimens with edge-shaped ends whereas all the other laboratories used the principle of pin-loading. BOLLENRATH et al[3] studied by means of photo-elastic models the effect of various specimen-end configurations on the stress distribution. According to these authors a smaller radius of fillet (0.25 in. instead of 0.5 in.) is more favourable when the distance between the pins and the gauge length remain constant. The same authors have also studied the stress distribution in specimens loaded by means of wedge-shaped ends. They reported that in this type of specimens bending stresses could not be easily avoided.

Concerning the methods for machining the specimens, Laboratory XI reported that the use of the electrical discharge method for preparing the pin holes or any other part of the specimens may result in abnormal fracture behaviour. It is thus considered useful to remove from the surface the material affected by electrical discharge machining.

The surface finish recommended in the specifications was the as delivered surface. It has been suggested that the measurement of the surface roughness would be desirable in order to better indentify the initial surface condition.

7.9.2 Pretest Inspection

The pretest inspection has been apparently adequate in all laboratories except for the evaluation of surface contamination by chemical analysis. The latter is actually delicate, expensive and time consuming. The limited results made available in this programme showed that a slight contamination in oxygen and nitrogen was occurring after testing at high temperature. For the TZM material and for the test conditions considered in this program it is far from obvious whether the test results have been affected by contamination. It is felt, however, that for refractory metals more sensitive to contamination, such as niobium or tantalum or for prolonged exposures at elevated temperature the control of contamination should be carried out.

7.9.3 Loading Apparatus and Methods

A variety of loading equipments were used by the participating laboratories. It is not clear whether factors relevant to the loading equipment such as precision and accuracy of load measurements, axiality, etc., have influenced the test results in a significant way. The low scatter in tensile data obtained by some of the participant laboratories reflects a very careful control of loading parameters amongst others. The exercise on the Vascojet 90 steel sheet has shown that the introduction of the vacuum chamber to the loading system did not alter significantly the average values of the results.

7.9.4 Strain Measurement and Strain Rate

Strain measurement through the cross head movement was used by several laboratories for the determination of the 0.2% offset yield strength. In no case has this method been used for the determination of elastic modulus.

A number of laboratories have successfully used extensometers up to the highest temperatures considered. Laboratory X developed an original type of extensometer where the elongation is measured along the neutral line of the specimens by means of extension arms in wire form. Laboratory IX measured the extension at the specimen ends. Laboratory XI used a commercially available extensometer where the elongation is measured at the gauge length. The latter laboratory has also evaluated the possibilities of an opto-electrical extensometer. It appears however that this type of extensometer requires more development work in order to render its operation reliable in high temperature testing. In a general way the cooperative programme has shown that strain measurements through extensometers is possible up to 1800°C provided that adequate equipment is used with the necessary care.

In Table 34 the average results of laboratories that have used an extensometer are compared with those of laboratories that have used the cross head movement as a method of strain measurement. In the case of the Vascojet steel (both testing conditions) and for the tests on TZM at 1050° and 1450°C the average yield strength obtained with an extensometer is slightly higher than that obtained without extensometer. For the tests on TZM at RT and at 1800°C the opposite tendency is observed however. Thus, from the data available in this programme it is not possible to conclude whether the yield strength is affected by measuring the strain from cross head motion or from an extensometer.

The calculation of strain from cross head movement data should, of course, be related to the actual length of the reduced section of the specimen and not to the nominal gauge length. It may also be necessary to consider also the participation to the deformation of a part of the fillet.

Data from the paper of BOLLENRATH et al[3] enable one to evaluate the fictitious gauge length. In this work, strain (ϵ_H) was measured over a distance H_0 = 50.8 mm including 28.4 mm for the reduced section length, 13 mm for the fillet, the remaining being a part of the specimen ends. Comparative measurements of strain were made with an extensometer over 25 mm of the gauge length (ϵ_M). In the elastic region the ratio ϵ_H: ϵ_M was found to be equal to 0.825. This corresponds to a fictitious gauge length of 41.9 mm which means that the contribution of the fillets and specimen ends to the overall elastic deformation is appreciable. In the plastic region the ratio ϵ_H: ϵ_M falls to 0.6 which corresponds to a fictitious gauge length of 30.5 mm. This means that a part of the fillet also intervenes in the plastic deformation. For fillets of high radius the proportion of the fillet intervening in the fictitious gauge length is still higher.

Similarly to the strain the strain rate also should be related to the actual length of the specimen, when a constant cross-head speed is used. As known[5], constant cross-head speed results in low strain rates which progressively increase when the deformation approaches the plastic region. It is only within the latter that the nominal strain rate is approximately obtained. In view of the sensitivity of molybdenum to strain rate it may be expected that the 0.2% YS values obtained with controlled strain rate might be higher than those obtained with constant cross-head speed.

Table 35 compares some YS results obtained with constant strain rate to those obtained with constant cross-head speed. While the variability is not systematically affected by the method of strain rate control, the YS values appear to be systematically slightly higher in the case of constant strain rate.

The recommended strain rates, i.e. 0.005 ±0.001 per minute up to 0.5% offset and 0.05 ±0.01 per minute beyond 0.5% offset appear to be adequate. The increase in strain rate beyond 0.5% offset was performed by most laboratories without any particular difficulty.

Total elongation measurements were made in most cases by means of marks on the specimens without any inconvenience such as fracture at one of the markings.

7.9.5 Temperature Measurement and Control

Table 7 shows that for increasing temperatures in the range of 1050 to 1800°C the following methods were chosen for the measurement of temperature:

- chromel-alumel thermocouples.
- Pt-PtRh thermocouples.
- Mo-W thermocouples.
- W-Ir thermocouples.
- W-WRe thermocouples.
- Optical pyrometers.

These methods used separately or in combination, provide reliable means for the temperature measurement. Laboratory II reported a variation of the emf output of the Pt-Pt 10% Rh thermocouple used at 1450°C with silica-insulators. This was corrected by changing the thermocouple after each test. Laboratory XI suggested that for a given temperature the thermocouple having the lowest temperature to mv output ratio should be chosen. This is particularly important in the case of the 1450°C temperature where recrystallization of the TZM is occurring. In this case W-WR thermocouples should be preferred to Pt-PtRh ones because of their higher mv output.

The temperature variation at any point within the gauge length of the test piece was specified not to vary by more than ±10°C for nominal temperatures of 1000°-1500°C, or by ±15°C for nominal temperatures in the range of 1500°-1900°C.

At least as good control as that specified has been achieved by most of the laboratories. Examination of the data in Table 7 shows that the permissible variations may be reduced by 50% and still allow most of the laboratories to be within the specified limits.

The temperature gradient along the gauge length was specified not to exceed 0.5% of the nominal test temperature in °C. The gradient depends strongly on the size of the furnace used and also on the cooling conditions of the pulling rods. In many instances the specified limits have been exceeded whereas in others the observed gradients were quite satisfactory. It was reported from some laboratories that the control of the temperature gradient became easier as the temperature increased. This behaviour is attributed to the lesser effect of the cooling in the pulling rods.

7.9.6 Heating Methods and Rate

Most of the laboratories have used radiant cold-wall furnaces fitted with molybdenum, tungsten or tantalum resistors. One laboratory used a silicon carbide furnace with an alumina tube, one used self-resistance heating and one an electron beam furnace.

Self-resistance heating is a well controlled method which for the sheet materials considered here gives comparable results provided the other conditions remain constant. The results of the cooperative program have confirmed that elongation values are significantly

low in the case of self-resistance heating. They have also shown that the low heating times which are preferred in self-resistance heating, result in large property variations when at the test temperature the alloy is subject to structural instability.

Furthermore, self-resistance heating was not considered adequate for test temperatures above 1450°C.

Apart from these remarks the 1050°C test results reported by laboratory IV that has used self-resistance heating do not appear to differ from those of other laboratories, although 2 in. gauge length specimens were used.

The results given by laboratory XI on the high temperature tensile properties of TZM obtained with an electron beam furnace revealed that at the highest test temperatures of 1450°C and 1800°C the elongation values were too low. For the particular furnace considered the length of the heated zone was presumably insufficient to account for the high elongation of the specimens. This inconvenience is related to the particular equipment used and not to the method itself. Other advantages or disadvantages of the method have not been reported.

Heating time to temperature is of great importance, as previosuly shown, mainly because it affects at certain temperatures the microstructure of the tested alloy. In this programme, the TZM sheet experienced recrystallization at 1450°C. The holding time at temperature which was specified at 15 minutes proved to be satisfactory for achieving a uniform temperature along the gauge length. The neating time to temperature was recommended to be not less than five minutes and not more than 60 minutes. In fact the actual temperature versus time variation varied according to the degassing rate during the earlier stages of heating. It appears appropriate to recommend a sufficient time for degassing up to 450°C and to specify a heating rate of 15-20°C per minute from 450°C to the test temperature. Such a procedure is justified by the fact that refractory metals are structurally stable up to 450°C.

7.9.7 Test Environment

Vacuum was proved to be the most reliable environment for high-temperature testing of uncoated refractory metals. A vacuum of better than 10^{-4} mm Hg was well controlled. For the molybdenum alloy considered in this programme and for the short periods involved in tensile testing, the vacuum recommended appears to be satisfactory. However for other refractory metals or for longer test periods a higher vacuum may be necessary to avoid contamination.

7.10 Modification of the Specifications

On the basis of the preceding discussion on test results it appears appropriate to modify some of the original recommendations given in Appendix A. The modifications proposed are summarized in Appendix D. They reflect the experience obtained from the Agard Cooperative Testing programme. Undoubtedly more stringent specifications may be drawn up if sophisticated equipment and/or extreme care is afforded in the carrying out of the tests.

7.11 Properties of the Tested Materials

In the previous chapters the variability of the test results within a laboratory or among several laboratories was discussed in terms of the test techniques and of the specifications used. Considering the latter as representing the actual state of the art, it is possible also to ascertain the properties of the material tested as an overall result of an interlaboratory investigation.

The results relevant to the Vascojet 90 steel sheet are shown in Table 45, whereas the results for the transverse properties of the TZM molybdenum alloy sheet are presented in Table 46. In these tables the average values have been computed on the basis of all the

results obtained in different laboratories. From the total group of data the standard deviation was computed. The latter was also expressed as a percentage of the average value. The standard deviations considered derive from total variances which include inter- as well as intralaboratory variances.

For the Vascojet 90 steel sheet the standard deviations for UTS and YS amount to 1.61 and 1.03 kg/mm^2 respectively which represent 2.5 and 2.02% of the corresponding overall values. These low standard deviations indicate, as previously stated, good homogeneity of the material and good interlaboratory control of test techniques and conditions. The above values of standard deviations are not significantly lower than that computed from 20 measurements carried out by Sud Aviation on the original sheet (Table 5).

The standard deviation for elongation amounts to 8.8% of the overall average value.

The absolute values for the standard deviations of UTS and YS of the TZM sheet decrease as the test temperature increased (Table 46). On a percent basis, however, the standard deviation increases with the temperature to reach, for example, 42% of 0.2% YS at 1800°C.

The variation with test temperature of the standard deviation for elongation is smaller when expressed in percent of the overall average value.

Average modulus values have been also computed and are reproduced in Tables 45 and 46 respectively for Vascojet and TZM sheet. The longitudinal properties of the TZM sheet have not been made the object of any overall computation since only a few laboratories gave such results. As previously discussed the data obtained on the longitudinal properties indicate a fairly good homogeneity of the TZM sheet in the two directions.

8. SUMMARY AND CONCLUSIONS

The interlaboratory cooperative programme, in which 11 laboratories from different NATO countries have participated, dealt with:

- the determination of room temperature testing properties of Vascojet 90 steel sheet, for conventional testing techniques.
- the same under high temperature testing conditions.
- the determination of the recrystallization temperature of TZM molybdenum alloy sheet.
- the determination of the tensile properties of TZM molybdenum alloys sheet at RT, 1050°, 1450° and 1800°C.

The objective of the programme was to generate information on the adequacy of test techniques and specifications and also on the properties of the material tested.

8.1 Effect of Test Techniques

The accuracy of mechanical properties measurements depends mainly on the accuracy with which the various test parameters are measured and controlled. If the effect of a test parameter error on the variation of the property measured is known, then the total variation arising from different errors can be evaluated. An assessment of this type is given in Appendix G by Bollenrath, Feldman and Happek. In this note the test parameters considered are strain, strain rate and temperature. The property evaluated is the 0.2% yield strength and creep rupture strength. In this note nomograms are presented which allow for the determination of the overall error on the basis of the individual errors.

Regarding the parameters related to the test techniques, the following observations can be made:

- one inch gauge length specimens proved to be satisfactory.
- two inch gauge length specimens affect the elongation values of TZM sheet which is subject to localized necking.

- pin loading proved to be generally satisfactory.
- mechanical or hydraulic loading systems proved to be equally satisfactory.
- extensometers of the mechanical-electrical type proved to be reliable up to 1800°C. They have been successfully associated with strain rate control. Opto-electrical extensometers deserve further development.
- modulus values were reported only from laboratories that used an extensometer.
- most of the laboratories approximated, in high temperature tests, constant strain rate by constant cross-head speed and strain measurement by cross-head movement.
- the comparison of data derived from tests where strain or strain rate was controlled through cross-head movement or speed to those where control occurred by means of an extensometer, was not conclusive.
- the widely used method for heating up to and exceeding 1800°C is radiant heating by means of tantalum or tungsten resistors located in cold wall furnaces. The size of the heating element strongly affects the temperature gradient along the specimens gauge length.
- self-resistance heating proved to be adequate up to 1450°C. It requires, however, 2 in. gauge length specimens. Furthermore, the short heating and holding times preferred with this type of heating results in non-comparable data in temperature ranges where structural instability may be encountered.
- an electron beam furnace was used successfully up to 1800°C. A shortcoming of the particular equipment used was inadequate temperature gradient control along the specimen's gauge length at temperatures at which high elongation occurs. No distinct advantages have been reported for this type of heating method.
- the results on the Vascojet 90 steel have shown that the introduction of the vacuum chamber in the loading line does not affect significantly the test results.
- temperature measurements up to 1800°C have been successfully performed by means of thermocouples and optical pyrometers. At increasing test temperature the thermocouples included: chromel-alumel, Pt-PtRh, W-Mo, W-Ir and W-WRe.
- temperature control versus time was satisfactory. Temperature gradient control was also satisfactory except in cases where furnaces of limited size were used.
- an environment consisting of a vacuum of better than 10^{-4} mm Hg appeared to be appropriate. The few data available indicate that only oxygen and to some extent nitrogen contaminated the TZM material during testing.

8.2 Adequacy of the Specifications

Broadly, a specification may be considered as adequate if it insures satisfactory intralaboratory and interlaboratory reproducibility. Examination of the overall data obtained in this programme indicated the adequacy of the specification used, in so far as the intralaboratory variability is concerned. Interlaboratory reproducibility was, however, in many instances rather poor. As shown from the considerations in Appendix C, the extent of the interlaboratory variability cannot be explained in terms of material inhomogeneity. This observation would entail as a conclusion that the specifications are not adequate to ensure acceptable interlaboratory reproducibility. It should be noticed, however, that deviations from the recommended procedures have occurred in many laboratories and have certainly contributed to the large interlaboratory variability. The quantitative assessment of the effect of testing variables appear to be feasible only by systematic tests carried out in one laboratory. In the light of the results of the present programme the design of such tests could be made more efficient. Their implementation should be encouraged considering the fact that they would provide the only factual background for a more meaningful modification of the specifications.

8.3 Properties of the Materials

Assuming that the methods and specifications used in the collaborative programme represent the current state of the art in this field it is possible to ascertain the properties of the materials tested on the basis of the collaborative evaluation. For example, on the basis of the data summarized in Tables 45 and 46 it is possible to determine the confidence limits of a test result obtained in any one of the laboratories. The 95% confidence limits may be approximated by adding to the grand average the quantity ±2.0 × standard deviation. The confidence limits of the average of n tests may be obtained similarly, except that the quantity added is divided by $\sqrt{n}$*.

These confidence limits for a single test result are represented graphically in Figures 11 to 13 for the transverse tensile properties of TZM sheet. The range of variation indicated includes, as stated, above, the intra as well as interlaboratory variability and also that originating from the material considered. The range for the strength properties and the modulus decreases with increasing temperature. It should however be noted that, as the strength level decreases, the relative variation becomes extremely large.

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* The more accurate determination of the confidence limits should make use of the values of $t\alpha$ in $t\alpha s/\sqrt{n}$ corresponding to the degrees of freedom used in the determination of s .

TABLE 1

Tests Considered in the Cooperative Programme

	Type of Tests	*No. of Tests*
TZM Mo Sheet	Tensile Test at RT Tensile Test at 1050°C Tensile Test at 1450°C Tensile Test at 1800°C Recrystallization Temperature	10 10 10 10 -
Vascojet 90 steel sheet	Conventional RT tensile tests RT Tensile Tests in vacuum	10 10

TABLE 2

List of Participating Laboratories

BELGIUM.

- Centre d'Etudes Nucléaires, Mol
- Centre National de Recherches Métallurgiques, Liège.

CANADA.

- Department of Mines and Technical Surveys, Ottawa.

FRANCE.

- Centre de Recherches Métallurgiques de Péchiney, Chambéry.
- Laboratoire Central de Sud Aviation, Courbevoie (Seine).

GERMANY.

- Institut für Werkstoffkunde, Technische Hochschule Aachen.

UNITED KINGDOM.

- Imperial Metal Industries (Kynoch) Ltd, Witton Birmingham 6.
- National Engineering Laboratory, East Kilbride, Glasgow.

UNITED STATES OF AMERICA.

- Air Force Materials Laboratory, Wright Patterson Air Force Base, Ohio.
- General Dynamics, Pomona, California.
- Naval Air Engineering Center, Philadelphia, 12, Pa.

TABLE 3

Summary of Work performed in each Laboratory on TZM Sheet

Laboratory	*Sheet No.*	*Recr.*	*RT*	*1050°*	*1450°*	*1800°*
I	16	Yes	Tr	Tr	Tr	-
IIa	17	-	Tr	Tr	Tr	Tr
IIb	18	Yes	Tr	-	-	-
III	14	Yes	Tr	Tr	Tr	Tr
IV	5 and 6	-	Tr	Tr	Tr	-
V	11	Yes	Tr	Tr	Tr	-
VI	12	Yes	Long.	Long.	Long.	Long.
VII	13	-	Tr	Tr	-	-
VIII	10	Yes	Tr	Tr	Tr	Tr
IX	1	Yes	Tr	Tr	Tr	Tr
X	9	Yes	Tr	Tr	Tr	Tr
XI	?	-	Tr	Tr	Tr	Tr
XIb	8	Yes	Long.	Long.	Long.	Long.

Tr: Transverse
Long.: Longitudinal

TABLE 4

Producer's Data on Tensile Properties at RT of TZM Sheet.

		Transverse		*Longitudinal*	
		End A	*End B*	*End A*	*End B*
Sheet 57	UTS	92.9	91.6	91.2	90.3
	YS	89.4	87.2	80.6	81.1
	EL	12.3	12.9	17.8	15.2
Sheet 58	UTS	94.8	94.4	90.3	86.10
	YS	89.6	88.9	78.9	78.70
	EL	13.0	13.1	14.5	18.70

TABLE 5

Homogenity of a Vascojet 90 Steel Sheet Assessed by RT Tensile Test Data.

	U.T.S. kg/mm^2	*0.2 Y.S. kg/mm^2*	*Elongation %*
Number of measures	20	20	20
Average	64.3	49.25	22.8
Max.	68.4	52.7	26.0
Min.	59.5	46.0	20.5
Range	8.9	6.7	5.5
Variance	4.2	2.4	2.25
Standard deviation	2.05	1.55	1.49

TABLE 6

Conditions for Tests At Room Temperature

	I	II	III	IV	V	VI	VII	VIII	IX	X	XI
Specimen	Normal (1")	Normal (1")	Normal (1")	Normal (2")	Normal (1")	Normal (1")	Normal (1")	Normal (1")	Normal 1") modified	Normal (1") modified	Normal (1")
Edge surface Finish	Mill	Longitudinal polishing 00	Longitudinal polishing 00	N.R.	As milled	Longitudinal polishing 00	Longitudinal polishing 00	Longitudinal polishing 00	Longitudinal polishing 00	Longitudinal polishing 00	Polishing 00
Tensile equipment	Universal Mechanical	Universal hydraulic and Mechanical	Universal Mechanical	Special hydraulic servo-controlled	Universal Hydraulic	Universal Mechanical	Universal Mechanical	Universal Mechanical	Universal Hydraulic	Universal Mechanical	Universal Mechanical
Strain measurements	Cross-head Motion	Extensometer	Cross-head Motion	Extensometer	Optical Extensometer	Extensometer	Extensometer	Cross-head Motion	Extensometer	Extensometer	Extensometer
Accuracy strain measurements	N.R.	4×10^{-5}	N.R	1×10^{-4}	5×10^{-5}	N.R.	1×10^{-5}	N.R	10^{-2}	2.5×10^{-5}	10^{-4}
Strain Rate	-	0.005 0.05	- -	0.005 0.05	0.004-0.005 0.06-0.10	-	-	-		0.005 0.06	0.005 0.05 } for TZM
Cross-head Speed	0.1 mm/min.	-	0.127 mm/min. 1.27 mm/min.	-	-	0.127 mm/min. 1.27 mm/min.	0.127 mm/min 127 mm/min.	0.5 mm/min.	0.127 mm/min. 127 mm/min.	-	0.127 1.27 } for Vasc.
Load measurements	Load Cell	Load Cell	Load Cell	Ring dynamometer	N.R.	Load Cell Daily Calibration	Load Cell	Load Cell	Mechanical	Torsion Bars	Load Cell
Accuracy load measurements	±0.5%	±0.5%	±0.5%	±0.5%	Grade A BS 1610:1964	N.R.	Grade A BS 1610:1964	±0.5%	N.R.	±0.5%	±0.5%
Elongation measurements	Markings on specimens (25.4 mm)	Markings on specimens (25.4 mm)	Minimum distance between pinholes*	N.R.	Markings on specimens (25.4 mm)	Overall length	Marks on specimen (25.4)mm)	Markings on specimens (25.4 mm)	N.R.	Markings on specimens (25.4 mm)	Marks on specimen (25.4 mm)
Atmosphere	Air	Air	Air	N.R.	Air	Air	Air	Vacuum	Air	Vascojet Air TZM: Vacuum	Air
Temperature	N.R.	25°C	25°C	N.R.	19-24°C: TZM 18-23°C: Vasc.	21-23°C	18-21°C	18-22°C	23 ± 2°C	22 ± 1°C	28-29°C (TZM)

N.R.: Non reported

* The specimens of Vascojet steel were provided with markings.

TABLE 7

Conditions for Tensile Tests of TZM Sheet at Elevated Temperature and of Vascojet 90 - Steel Sheet at RT (Partly)

	I	II	III	IV	V	VI
Specimen	Normal (1")	Normal (1")	Normal (1")	Special (2")	Normal (1")	Normal (1")
Edge surface Finish	Mill	Longitudinal polishing 00	Longitudinal polishing 00	N.R.	As milled	Longitudinal polishing 00
Tensile equipment	Universal Mechanical	Universal Mechanical	Universal Mechanical	Special-Hydraulic Servo-Controlled	Modified Stress-rupture machine	Universal Mechanical
Strain Measurement	Cross-head Movement	Cross-head Movement	Cross-head Movement	Extensometer	Cross-head Movement	Cross-head Movement
Accuracy Measurements	N R	N.R	N R	1 10^{-4}	N.R	N.R
Strain-Rate	-	-	-	up to 0.5% 0.005 from 0.5% to rupture. 0.05	-	-
Cross-head Speed	0 1 mm/min	0 2 mm/min. 2 mm min	0 127 mm/min 1 27 mm/min	-	0 127 mm/min 1 27 mm/min.	0 127 mm/min 1.27 mm/min.
Load Measurement	Load Cell	Load Cell	Load Cell	Ring dynamometer	Proving ring	Load Cell Daily-Calibration
Accuracy load Measurement	± 0 5%	± 0 5%	± 0 5%	± 0 5%	N R	N R.
Heating Method	Crusilite Resistors	Tantalum Resistors	Tantalum Resistors	Self Resistance	Ta Resistors	Tantalum Resistors
Heating time to Temperature	90 min at 1050°C 120 min at 1450°C	45 min at 1050°C 60 min. at 1450°C and 1800°C	45-60 min	62 sec at 1050°C 76 sec at 1450°C	Preheating to 450°C overnight 75 min at 1050°C 105 min. at 1450°C.	Up to 1000°C according to Degassing above 1000°C 60°C/min.
Time at Temperature	15 min	15 min	15 min.	3 min.	15 min.	15 min.
Temperature Measurements	Pt/Pt 10 Rh 1050°C Pt5 Rh/Pt20Rh 1450°C	Pt/Pt 10 Rh at 1050°C and 1450°C Mo-W at 1800°C	Optical Pyrometer	Thermocouple	Pt/Pt 13Rh Thermocouple	up to 1450°C Pt-Pt10 Rh 1800°C: Optical Pyrometer
Temperature Variation	± 3°c	± 5°C	N R	± 5 5°C	1054 ± 3°C 1446 ± 6°C	N.R.
Temperature Variation along specimen	N R.	1050°C · 4°C 1450°C 3°C 1800°C. N R	N R	N.R.	1050°C 15°C 145[illegible] 10°C	1050°C + 0.5% 1450°C: and 1800°C ± 0.5%
Atmosphere	Vacuum 10^{-4} 2 × 10^{-5}	Vacuum 7 × 10^{-5} - 10^{-5}	Vacuum 10^{-4} or better	Vacuum 2 5 - 4 × 10^{-2}	Vacuum 2 × 10^{-4} - 4 × 10^{-5}	Vacuum 10^{-4} or better
Elongation Measurements	Markings on specimen (25 4 mm)	Markings on specimen (25 4 mm)	Minimum distance between pinholes*	N.R.	Markings on specimens (25.3 mm)	Overall length

	VII	VIII	IX	X	XI
Specimen	Normal (1")	Normal (1") modified	Normal (1") modified	Normal (1") modified	Normal (1")
Edge surface Finish	Longitudinal polishing 00	Longitudinal polishing 00	Longitudinal polishing 00	Longitudinal polishing 00	Polishing 00
Tensile equipment	Universal Mechanical	Universal Mechanical	Mechanical	Universal Mechanical	Universal Mechanical
Strain Measurement	TZM cross-head vasco: entenso.	Cross-head Movement	Extensometer	Extensometer	Extensometer
Accuracy Measurements	N.R.	N.R.	10^{-2}	2.5 × 10^{-5}	10^{-1}
Strain-Rate	-	-	-	0 005 0 05	-
Cross-head Speed	0.127 mm/min 1.27 mm/min	0 5 mm/min	0.127 mm/min. 1.27 mm/min.	-	0.127 mm/min. 1.27 mm/min.
Load Measurement	Load Cell	Load Cell	Load Cell	Torsion Bars	Load Cell
Accuracy load Measurement		± 0.5%	1%	± 0 5%	± 0.5%
Heating Method	Mo-resistors	Tantalum Resistors	Tantalum Resistors	Tungsten Resistors	Ta.Res. (Sh.7) El.Beam (Sh.8)
Heating time to Temperature	Preheat to 450°C 60 min. to temperature	30 min.	20-30 min	60 min.	1050°: 20 min. 1150°: 25 min. 1800°. 30 min.
Time at Temperature	15 min.	15 min	15 min.	15 min.	1050°: 15 min. 1450°: 40 min. 1800°: 20 min.
Temperature Measurements	Pt/Pt 13Rh thermocouple	Pt 20 Rh Pt 40Rh up to 1450°C/WRe/ W26Re at 1800°C	Pt/Pt/Rh up to 1450°C optical Pyrometer at 1800°C	Pyrometer and Pt/PtRh up to 1450°C WRe 3/ WRe26 at 1800°C	1050°: chromel-alumel 1450 and 1500°C W3Re/W26Re
Temperature Variation	1050°C ± 8°C	1050°C and 1450°C ± 3°C 1800°C ± 5°C	N.R.	± 2°C	1050°C ± 3°C max 1450° - 1800°C ± 5°C.
Temperature Variation along specimen	1050°C: 10-40°C	1050°C ± 1°C 1450°C ± 2°C 1800°C ± 3°C	0.5% max.	1050°C ± 4.5°C 1450°C ± 3°C 1800°C ± 3°C	± 7°C
Atmosphere	10^{-4}	Vacuum 4 × 10^{-5} or better	10^{-4}	10^{-4} - 2 × 10^{-5}	6 × 10^{-5}
Elongation Measurements	Marks on specimen (25.4 mm)	Markings on specimens (25.4 mm)	N.R.	Markings on specimens 25.4 mm)	Marks on specimen (25.4)

N.R. Non reported

*Markings in Vasco 90 specimens.

TABLE 8

Room Temperature Conventional U.T.S. of Vascojet 90 Steel Sheet.

Test No.	I	II	III	V	VI	VII	VIII	X	XI
1	64.4	65.1	65.7	64.6	68.3	64.6	63.5	65.3	64.1
2	64.1	63.4	66.4	64.1	68.3	66.2	63.8	64.3	62.8
3	62.1	64.8	66.7	64.9	67.3	66.0	63.9	65.4	63.1
4	62.1	63.9	66.9	64.9	66.2	65.0	63.6	64.9	62.9
5	64.4	64.3	65.4	64.9	67.5	63.6	63.0	64.4	64.1
6	61.8	64.6	66.3	65.2	65.9	66.4	63.0	64.4	62.9
7	60.1	63.2	65.2	65.0	67.1	64.0	64.0	65.5	63.9
8	61.2	63.6	66.3	65.0	65.7	64.0	63.2	65.1	62.4
9	62.2	63.5	66.3	65.0	66.4	64.0	63.2	65.1	62.3
10	64.0	63.9	67.2	65.5	67.8	64.7	63.4	64.2	61.8
Av.	62.6	64.0	66.2	64.9	67.1	64.8	63.5	64.9	63.7.
Max	64.4	65.1	67.2	65.5	68.3	66.4	64.0	65.5	64.1
Min	60.1	63.2	65.2	64.1	65.9	63.6	63.0	64.2	61.8
R	4.3	1.9	2.0	1.4	2.4	2.8	1.0	1.3	2.3
V	2.24	0.41	0.41	0.13	0.75	1.04	0.13	0.24	2.89
S	1.5	0.64	0.64	0.36	0.87	1.02	0.36	0.49	1.70

TABLE 9

Room Temperature Conventional 0.2% Y.S. of Vascojet 90 Steel Sheet

Test No.	I	II	III	V	VI	VII	VIII	X	XI
1	51.6	50.3	51.5	49.6	51.4	49.7	50.3	50.4	49.2
2	47.2	49.9	51.0	49.6	51.1	51.1	50.2	50.9	49.1
3	47.9	50.2	51.0	51.0	51.1	50.7	50.6	50.3	48.6
4	48.7	53.2	51.5	50.1	50.6	50.3	50.3	50.6	49.3
5	51.5	50.5	49.8	50.1	50.4	49.3	49.3	50.6	49.5
6	47.2	50.2	50.6	50.6	50.0	51.1	50.7	50.9	49.3
7	49.5	49.5	49.4	50.6	50.9	50.3	51.1	51.5	50.1
8	47.0	49.7	50.7	50.7	49.5	50.4	50.5	51.4	48.9
9	48.6	49.5	49.9	50.9	50.6	50.3	50.0	50.8	48.9
10	51.0	50.7	51.0	51.3	50.9	50.8	50.0	51.5	48.7
Av.	49.0	50.4	50.6	50.4	50.7	50.4	50.3	50.9	49.2
Max	51.6	53.2	51.5	51.3	51.4	51.1	51.1	51.5	50.1
Min	47.0	49.5	49.4	49.6	49.5	49.3	49.3	50.3	48.6
R	4.6	3.7	2.1	1.7	1.9	1.8	1.8	1.2	1.5
V	3.24	1.15	0.51	0.33	0.32	0.32	0.23	0.19	0.19
S	1.80	1.07	0.72	0.58	0.56	0.57	0.48	0.44	0.43

TABLE 10

Room Temperature Conventional Elongation of Vascojet 90 Steel Sheet

Test No.	*I*	*II*	*III*	*V*	*VI*	*VII*	*VIII*	*X*	*XI*
1	19.6	18.8	18.0	21.0	18.0	22.0	17.7	19.1	28.8
2	22.4	18.7	21.0	22.0	15.0	19.0	19.7	18.8	19.6
3	19.6	21.1	20.0	22.0	17.0	20.0	15.8	19.3	18.5
4	19.6	19.8	19.0	21.0	17.0	21.0	17.7	18.5	16.7
5	19.6	21.5	16.0	19.0	17.0	2[illegible].0	17.7	19.1	17.5
6	18.8	21.2	19.0	21.0	19.0	22.0	19.7	19.0	16.0
7	18.5	21.2	19.0	22.0	18.0	20.0	19.7	19.0	17.5
8	19.6	18.8	19.0	19.0	17.0	20.0	17.7	18.2	17.5
9	20.0	20.2	18.0	21.0	16.0	20.0	19.7	20.9	17.5
10	22.0	16.2	17.0	19.0	17.0	20.0	17.7	17.1	17.3
Av.	20.0	19.8	18.6	20.7	17.1	20.6	18.3	18.9	17.7
Max.	22.4	21.5	21.0	22.0	19.0	22.0	19.7	20.9	19.6
Min.	18.5	16.2	16.0	19.0	15.0	19.0	15.8	17.1	16.0
R	3.9	5.3	5.0	3.0	4.0	3.0	3.9	3.8	3.6
V	1.58	2.75	2.04	1.56	1.21	1.15	1.76	0.90	1.07
S	1.25	1.66	1.42	1.25	1.10	1.07	1.32	0.95	1.035

TABLE 11

Conventional Elastic Modulus ($10^3 kg/mm^2$) of Vascojet 90 Steel Sheet

Test No.	*II*	*X*	*XI* (a)	*XI* (b)
1	19.90	21.6	22.6	22.5
2	21.40	20.9	23.3	21.5
3	20.80	19.7	20.5	23.5
4	19.00	20.8	20.4	21.8
5	22.70	20.8	21.9	20.9
6	20.50	21.6	21.1	
7	19.20	21.2	21.4	
8	20.40	21.4	21.6	
9	18.10	20.9	21.4	
10	21.00	20.4	21.0	
Av.	20.30	20.9	21.5	22.0
Max.	22.70	21.6	23.3	23.5
Min.	18.10	19.7	20.4	20.9
R	4.60	1.9	2.9	2.6
V	1.74	0.33	0.81	1.0
S	1.31	0.57	0.89	1.0

(*a*) V-grip loading
(*b*) Pin loading.

TABLE 12

RT U.T.S. of Vascojet 90 Steel Sheet under High Temperature Testing Conditions

Test No.	I	II	III	IV	V	VI	VII	VIII	X	XI
1	64.8	64.2	62.8	68.6	65.7	68.4	64.0	63.4	63.3	68.1
2	64.4	64.0	63.4	68.4	65.8	68.2	64.7	62.1	63.8	65.5
3	64.5	64.3	65.2	69.2	65.7	66.8	63.2	62.3	64.6	64.5
4	64.1	63.4	65.3	69.3	66.1	67.1	64.0	62.9	64.3	63.1
5	64.7	64.1	65.4	69.2	65.7	66.2	64.7	63.8	65.5	65.1
6	64.8	65.0	65.5	69.8	65.8	67.4	63.8	62.8	64.2	67.3
7	65.1	63.6	66.2	68.9	65.8	68.7	63.7	62.6	63.0	64.5
8	64.8	64.0	65.5	69.2	65.8	68.9	65.4	62.8	64.8	65.3
9	64.2	63.8	66.5	69.5	65.8	68.9	64.3	62.4	63.8	66.3
10	63.0	64.0	66.9	68.8	66.9	68.7	63.2	63.0	64.7	66.2
Av.	64.4	64.0	65.3	69.1	65.9	67.9	64.1	62.8	64.2	65.6
Max.	65.1	65.0	66.9	69.8	66.9	68.9	65.4	63.8	65.5	68.1
Min.	63.0	63.4	62.8	68.4	65.7	66.2	63.2	62.1	63.0	63.1
R	2.1	1.6	4.1	1.4	1.2	2.7	2.2	1.7	2.5	5.0
V	0.34	0.18	1.64	0.17	0.13	0.95	0.48	0.26	0.56	2.11
S	0.59	0.43	1.28	0.42	0.36	0.97	0.69	0.51	0.74	1.45

TABLE 13

RT 0.2% YS of Vascojet 90 Steel Sheet under High Temperature Testing Conditions

Test No.	I	II	III	IV	V	VI	VII	VIII	X	XI
1	-	50.6	48.1	52.9	49.8	51.0	49.6	50.5	50.8	52.0
2	48.7	49.5	49.1	53.4	49.3	51.7	49.9	49.5	49.8	49.3
3	49.3	50.5	50.1	53.3	49.8	51.0	47.1	48.3	50.6	50.2
4	48.8	49.5	48.9	53.7	50.6	50.5	48.9	48.0	50.2	49.6
5	48.1	49.7	50.5	53.9	50.4	49.9	50.1	49.8	51.0	50.2
6	48.4	51.8	49.4	54.1	50.2	50.7	49.7	49.1	51.1	
7	48.4	49.4	50.3	53.8	49.9	52.8	49.6	47.7	50.9	50.0
8	48.7	49.6	49.3	53.9	50.7	51.6	50.5	48.9	51.6	50.8
9	48.4	49.7	50.6	54.6	49.9	51.9	50.3	48.2	51.6	51.9
10	48.8	50.8	51.4	53.5	51.7	51.7	49.2	49.5	51.0	54.8
Av.	48.6	50.1	49.8	53.7	50.2	51.3	49.5	48.9	50.9	50.9
Max.	49.3	51.8	51.4	54.3	51.7	52.8	50.5	50.5	51.6	54.8
Min.	48.1	49.4	48.1	52.9	49.3	49.9	47.1	47.7	49.8	49.3
R	1.2	2.4	3.3	1.4	2.4	2.9	3.4	2.8	1.8	5.5
V	0.12	0.61	0.95	0.17	0.44	0.68	0.93	0.80	0.30	2.92
S	0.34	0.78	0.97	0.41	0.66	0.82	0.96	0.89	0.55	1.71

TABLE 14

RT Elongation of Vascojet 90 Steel Sheet under High Temperature Conditions

Test No.	I	II	III	IV	V	VI	VII	VIII	X	XI
1	18.9	23.4	18.0	16.5	21.0	16.0	21.0	17.7	18.2	18.8
2	18.9	18.7	21.0	16.5	20.0	17.0	20.0	17.7	16.8	19.6
3	18.9	20.8	19.0	16.5	18.0	18.0	20.0	17.7	18.2	17.9
4	18.5	18.9	20.0	17.0	18.0	17.0	21.0	15.8	18.5	14.8
5	19.7	18.7	19.0	17.5	19.0	15.0	20.0	19.7	18.8	15.6
6	20.5	22.2	20.0	17.0	19.0	17.0	19.0	17.7	18.1	16.2
7	19.7	17.1	19.0	17.5	18.0	-	19.0	19.7	18.1	16.3
8	18.9	21.4	19.0	18.0	20.0	18.0	20.0	17.7	18.1	17.0
9	19.7	19.5	18.0	14.0	18.0	16.0	20.0	17.7	16.3	17.2
10	19.7	20.0	16.0	17.0	20.0	16.0	20.0	17.7	19.4	16.9
Av.	19.3	20.1	18.9	16.7	19.1	16.7	20.0	17.9	18.1	17.1
Max.	20.5	23.4	21.0	18.0	21.0	18.0	21.0	19.7	19.4	19.6
Min.	18.5	17.1	16.0	14.0	18.0	15.0	19.0	15.8	16.3	14.8
R	2.0	6.3	5.0	4.0	3.0	3.0	2.0	3.9	3.1	4.8
V	0.37	3.57	1.87	1.18	1.21	1.00	0.44	1.24	0.80	2.03
S	0.60	1.89	1.37	1.08	1.10	1.00	0.66	1.11	0.89	1.42

TABLE 15

RT Elastic Modulus ($10^3 kg/mm^2$) of Vascojet 90 Steel Sheet under High Temperature Testing Conditions

Test No.	*IV*	*X*	*XI*
1	21.1	20.5	-
2	20.5	21.5	23.9
3	20.5	21.1	19.2
4	20.8	21.5	21.1
5	20.7	20.7	21.8
6	20.8	21.0	22.6
7	20.6	20.8	25.0
8	20.7	20.8	21.2
9	21.6	20.4	23.8
10	21.4	21.1	21.6
Av.	20.9	20.9	22.2
Max.	21.6	21.5	25.0
Min.	20.5	20.4	19.2
R	1.1	1.1	5.8
V	0.14	0.14	3.14
S	0.37	0.37	1.77

TABLE 16

Recrystallization Temperature

Laboratory	*Sheet No.*	*Recrystal. Temp. °C.*	*Initial Hardness*	*Final Hardness*	*Hardness at Recrystallization Temp.*
I	16 (62)	1325	282	172	205
II	18 (62)	1335	307	175	220
V	11 (62)	1360 +10	308	192	230
VI	12 (62)	1371	300	155	180
VII	13 (62)	1346 - 1350	302	206	231
VIII	10 (62)	1375 +10	305	179	220
IX	1 (60)	1300 - 1350	317	190	214 - 239
X	9 (60)	1352	289	186	220
XI	8 (60)	1373	300	187	220
Producer	57 end A.	1343			
	57 end B.	1399			
	58 end A.	1371			
	58 end B.	1343			

TABLE 17

UTS at RT of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II(a)*	*II(b)*	*III*	*IV*	*V*	*VI(c)*	*VII*	*VIII*	*IX*	*X*	*XI(d)*	*XI(e)*
1	86.3	88.5	92.0	81.9	88.6	91.8	90.7	91.4	91.7	94.0	88.7	81.3	89.4
2	85.0	87.3	89.9	83.7	94.8	91.7	92.1	87.9	90.4	91.0	92.0	83.9	90.1
3	84.0	87.5	86.4		90.8	91.0	92.1	87.1	88.8	92.8	90.2	86.2	86.2
4	83.9	86.9	89.3		94.4	90.7	90.7	88.5	85.2	90.0	88.7	87.3	93.9
5	81.2	86.7	91.0		94.1	89.1	91.4	88.0	88.9		89.8	85.8	87.6
6	82.1	87.3	87.5		92.0	88.8	92.8	87.2	90.0		91.5	85.0	87.0
7	81.9	85.7	90.0		92.7	89.8	92.1	89.2	87.0		91.0	84.7	89.2
8	81.0	87.7	90.3		91.5	87.7	92.1	89.5	95.3		92.2	86.7	88.4
9	84.4	88.4			86.4	88.8	92.8	89.2	97.8		89.5	89.1	94.8
10	83.8	87.8			91.1	89.8	92.8	92.0	96.9		90.2	86.7	89.3
11					89.7								
Av.	83.4	87.4	89.5	82.8	91.5	89.9	92.1	89.0	91.2	91.9	90.4	85.7	89.6
Max.	86.3	88.5	92.0	83.7	94.8	91.8	92.8	92.0	97.8	94.0	92.2	89.1	94.8
Min.	81.0	85.7	86.4	81.9	86.4	87.7	90.7	87.1	85.2	90.0	88.7	81.3	86.2
R	5.3	2.8	5.6	1.8	8.4	4.1	2.1	4.9	12.6	4.0	3.5	7.8	8.6
V	3.03	0.68	3.31	1.62	6.60	1.85	0.65	2.71	17.76	3.21	1.60	4.49	7.76
S	1.74	0.82	1.81	1.27	2.57	1.36	0.81	1.64	4.21	1.79	1.26	2.12	2.79

(*a*) Results obtained with mechanical equipment, sheet No. 17

(*b*) Results obtained with hydraulic equipment, sheet No. 18

(*c*) Longitudinal properties, sheet No. 12

(*d*) Transverse properties, sheet No. 7

(*e*) Longitudinal properties, sheet No. 8.

TABLE 18

0.2% YS at RT of TZM Molybdenum Alloy Sheet

Test No	*I*	*II(a)*	*II(b)*	*III*	*IV*	*V*	*VI(c)*	*VII*	*VIII*	*IX*	*X*	*XI(d)*	*XI(e)*
1	74.9	75.8	74.6	68.9	83.3	76.9	80.1	77.0	82.5	76.0	75.1	71.9	83.7
2	73.5	75.7	73.0	71.7	86.3	76.4	80.8	75.2	79.9	76.3	76.8	72.0	84.6
3	73.9	76.5	72.3		85.1	75.6	80.8	73.0	79.0	77.0	76.3	74.5	79.3
4	74.1	76.8	76.4		85.3	75.0	80.1	75.9	74.5	76.5	75.3	77.1	87.2
5	72.3	75.1	81.9		84.5	75.7	80.1	74.5	80.1		75.0	71.7	82.2
6	72.6	75.6	76.4		80.7	75.0	81.5	73.8	80.8		75.5	72.3	81.3
7	74.5	77.0	79.5		–	75.1	80.1	75.9	79.3		75.8	70.5	81.0
8	71.6	77.0	73.0		84.1	73.4	80.8	75.9	82.5		76.3	72.6	83.4
9	74.7	76.7			84.0	73.5	80.8	75.2	82.4		75.3	72.4	86.8
10	74.1	74.7			85.8	75.7	80.8	79.4	83.0		75.6	74.2	80.3
11					82.5								
Av.	73.6	76.1	75.9	70.3	84.2	75.2	80.8	75.6	80.4	76.4	75.7	72.9	83.0
Max.	74.9	77.0	81.9	71.7	86.3	76.9	81.5	79.4	83.0	77.0	76.8	77.1	87.2
Min.	71.6	74.7	72.3	68.9	80.7	73.4	80.1	73.0	74.5	76.0	75.0	70.5	79.3
R	3.3	2.3	9.6	2.8	5.6	3.5	1.4	6.4	8.5	1.0	1.8	6.6	7.9
V	1.22	0.67	11.56	3.92	2.78	1.24	0.27	3.13	6.45	0.17	0.35	3.51	9.98
S	1.10	0.82	3.40	1.98	1.66	1.11	0.52	1.77	2.54	0.42	0.59	1.87	3.16

(*a*) (*b*) (*c*) (*d*) (*e*): See note Table 17.

TABLE 19

Elongation at RT of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II(a)*	*II(b)*	*III*	*IV*	*V*	*VI(c)*	*VII*	*VIII*	*IX*	*X*	*XI(d)*	*XI(e)*
1	16.1	19.6	20.6	2.0	8.5	18.0	7.0	18.0	18.7	14.0	16.2	14.9	13.4
2	15.3	19.5	–	10.0	8.7	19.0	4.0	20.0	17.7	16.0	13.8*	11.2	(*)
3	16.4	19.4	19.5		11.0	18.0	8.0	20.0	18.3	22.0	16.8	19.0	(*)
4	18.8	18.0	14.7		8.0	19.0	9.0	17.5	18.5	18.0	20.0	18.1	11.4
5	13.8	19.2	17.7		10.1	17.0	14.0	21.5	16.7		14.2	14.5	11.1
6	15.1	19.5	20.4		11.2	19.0	9.0	17.0	15.7		15.3	15.3	12.6
7	14.4	18.2	16.4		9.2	17.0	11.0	20.0	17.9		20.9	18.4	11.3
8	14.4	19.1	17.6		9.0	20.0	10.0	19.0	19.3		15.4	10.2	9.3
9	14.4	22.8			2.1	16.0	10.0	18.0	20.0		13.0*	13.9	10.6
10	15.3	20.4			10.7	19.0	10.0	16.5	19.7		15.4	15.3	11.4
11					8.6								
Av.	15.4	19.6	18.1	6.0	8.8	18.2	9.0	18.7	18.2	17.5	16.1	15.1	11.4
Max.	18.8	22.8	20.6	10.0	11.2	20.0	14.0	21.5	20.0	22.0	20.9	19.0	13.4
Min.	13.8	18.0	14.7	2.0	2.1	16.0	4.0	16.5	15.7	14.0	13.0	10.2	9.3
R	5.0	4.8	5.9	8.0	9.1	4.0	10.0	5.0	4.3	8.0	7.9	8.8	4.1
V	2.08	1.76	4.72	32.0	6.18	1.51	6.89	2.56	1.76	11.66	6.54	8.47	1.51
S	1.44	1.32	2.17	5.66	2.49	1.22	2.62	1.60	1.32	3.41	2.55	2.91	1.23

(*a*) (*b*) (*c*) (*d*) (*e*): See note Table 17.

(*) Specimens failed outside of gauge section.

TABLE 20

Elastic Modulus at RT of TZM Molybdenum Alloy Sheet
$10^3 kg/mm^2$

Test No.	*II*	*IV*	*VI(a)*	*X*	*XI(b)*	*XI(c)*
1	30.00	28.7	23.9	30.5	27.8	30.9
2	28.70	29.7	25.2	30.4	28.1	29.6
3	33.40	29.8	25.4	30.3	28.8	28.5
4	29.70	30.4	24.8	31.7	28.4	32.1
5	28.40	30.7	24.2	30.7	27.8	30.4
6	25.20	30.4	25.8	30.1	30.2	29.2
7	34.50	-	25.9	30.3	31.1	30.4
8	35.10	30.0	25.0	31.1	30.0	30.7
9	34.60	30.5	26.6	30.3	31.6	30.4
10	23.80	30.6	25.8	30.2	32.0	30.1
Av.	30.34	30.1	25.2	30.6	29.6	30.2
Max.	35.10	30.7	26.6	31.7	32.0	32.1
Min.	23.80	28.7	23.9	30.1	27.8	28.5
R	11.30	2.0	2.7	1.6	4.2	3.6
V	15.98	0.48	0.68	0.24	2.59	0.97
S	3.99	0.69	0.83	0.49	1.61	0.985

(*a*) Longitudinal direction

(*b*) Transverse direction sheet No.7

(*c*) Longitudinal direction, sheet No.8.

TABLE 21

UTS at 1050°C of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II*	*III*	*IV*	*V*	*VI(a)*	*VII*	*VIII*	*IX*	*X*	*XI(b)*	*XI(c)*
1	53.5	55.2	55.7	54.2	55.3	62.6	62.7	57.8	60.5	55.8	56.6	46.8
2	52.7	55.2	54.3	51.6	54.5	60.7	59.5	55.8	58.1	56.6	58.4	46.3
3	55.6	55.0	54.3	54.0	54.6	60.7	60.0	55.8	58.8	56.2	56.0	55.3
4	53.5	53.0	54.7	51.9	55.6	59.1	61.3	58.5	60.8	57.3	57.1	44.0
5	52.4	53.8	54.3	52.3	55.1	58.6	61.3	53.3	60.6	56.0	55.3	43.4
6	53.5	53.3	54.8	50.2	55.0	59.1	60.1	57.5		56.4	46.1	42.9
7	55.3	53.4		53.2	55.7	60.7	57.5	54.4		55.3	53.4	43.8
8	55.9	54.4		54.6	55.3	58.2	58.7	57.6		57.3	55.6	
9*	54.6	53.1		53.8	54.8	58.6	57.1	57.7		56.3	55.0	
10*	54.4	53.2		53.5	55.9	59.2	60.0	52.6		57.3		
11							59.0					
Av.	54.0	54.0	54.7	52.9	55.2	59.8	59.7	56.1	59.8	56.5	54.8	46.0
Max.	55.9	55.2	55.7	54.6	55.9	62.6	62.7	58.5	60.8	57.3	58.4	55.3
Min.	52.4	53.0	54.3	50.2	54.5	58.2	57.1	52.6	58.1	55.[illegible]	46.1	42.9
R	3.5	2.2	1.4	4.4	1.4	4.4	5.6	5.9	2.7	2.0	12.3	12.4
V	1.83	0.82	0.29	1.93	0.22	1.89	2.75	4.28	1.50	0.46	12.68	18.7
S	1.35	0.905	0.54	1.39	0.47	1.37	1.66	2.07	1.22	0.68	3.56	4.33

(*a*) Longitudinal

(*b*) Transverse

(*c*) Longitudinal

(*) Increase in Strain rate after 0.5% Strain.

TABLE 22

0.2% Y.S. at 1050°C of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II*	*III*	*IV*	*V*	*VI*	*VII*	*VIII*	*IX*	*X*	*XI*
1	-	43.8	49.9	49.4	48.3	44.3	55.7	47.5	45.3	49.2	49.5
2	44.3	39.1	42.2	49.2	46.3	50.6	53.2	49.8	44.4	49.6	49.3
3	45.9	43.7	42.5	50.0	47.2	53.5	53.2	48.7	44.1	49.2	48.6
4	44.4	44.2	47.4	47.9	48.2	53.8	56.1	53.5	44.7	50.8	49.8
5	43.6	40.8	48.5	48.9	47.7	53.8	52.5	48.6	44.0	48.4	47.7
6	39.6	42.5	47.4	47.2	47.7	53.8	54.2	54.6		49.8	43.8
7	-	45.5		49.3	46.1	49.3	52.6	50.4		50.6	47.7
8	43.2	38.8		49.6	48.3	52.9	53.2	50.0		50.6	47.9
9	43.2*	44.3		50.1	46.6	48.8	50.9	50.1		50.0	47.3
10	42.9*	48.0		49.9	46.5	51.4	52.3	48.3		49.9	
11							51.7				
Av.	43.5	43.1	46.3	49.1	47.3	51.2	53.2	50.2	44.5	49.8	48.0
Max.	45.9	48.0	49.9	50.1	48.3	53.8	56.1	54.6	45.3	50.8	49.8
Min.	39.6	38.8	42.2	47.2	46.1	44.3	50.9	47.5	44.0	48.4	43.8
R	6.3	9.2	7.7	2.9	2.2	9.5	5.2	7.1	1.3	2.4	6.0
V	4.49	8.15	10.29	0.87	0.74	9.54	2.48	5.13	0.27	0.56	3.23
S	2.12	2.85	3.20	0.93	0.86	3.1	1.57	2.26	0.52	0.74	1.79

(*a*) Longitudinal

(*) Increase in Strain rate after 0.5% Strain.

TABLE 23

Elongation at 1050°C of TZM Molybdenum Alloy Sheet

Test No.	I	II	III	IV	V	VI(a)	VII	VIII	IX	X	XI(b)	XI(c)
1	7.5	5.2	6.0	3.5	6.0	7.0	6.0	3.9	6.9	5.6	4.5	3.7
2	8.7	6.3	6.0	2.5	7.0	5.0	6.0	5.9	4.7	6.6	7.5	7.2
3	6.7	6.6	6.0	3.0	5.0	3.0	7.0	7.9	4.3	6.2	3.9	2.0
4	7.5	7.8	6.0	3.5	7.0	2.0	5.0	7.0	5.1	6.3	4.7	4.6
5	7.9	7.3	6.0	5.5	6.0	4.0	8.0	7.9	4.7	6.0	7.1	6.3
6	6.7	7.7	8.0	3.5	7.0	6.0	8.0	7.9		5.4	5.5	8.1
7	6.3	6.6		4.0	7.0	7.0	5.0	5.9		5.5	7.0	7.7
8	6.0	8.5		5.0	7.0	5.0	6.0	5.9		5.5	4.0	
9	6.7	7.8		4.5	5.0	7.0	5.0	6.3		5.5	4.9	
10	6.7	7.1		3.0	6.0	7.0	7.0	7.5		6.3		
11							5.0					
Av.	7.2	7.1	6.3	3.8	6.3	5.0	6.2	6.6	5.1	5.9	5.5	5.6
Max.	8.7	8.5	8.0	5.5	7.0	7.0	8.0	7.9	6.9	6.6	7.5	8.1
Min.	6.0	5.2	6.0	2.5	5.0	2.0	5.0	3.9	4.3	5.4	3.9	2.0
R	2.7	3.3	2.0	3.0	2.0	5.0	3.0	4.0	2.6	1.2	3.6*	6.1
V	0.80	0.89	0.66	0.9	0.67	3.4	1.36	1.65	1.04	0.19	1.95	5.21
S	0.89	0.94	0.81	0.95	0.82	1.85	1.16	1.28	1.02	0.43	1.39	2.29

(a) Longitudinal

(b) Transverse

(c) Longitudinal

(*) Increase in Strain rate after 0.5% Strain.

TABLE 24

Elastic Modulus at 1050°C of TZM Molybdenum Alloy Sheet
1000 kg/mm²

Test No.	IV	X	XI
1	22.5	26.8	22.1
2	22.2	26.7	23.9
3	23.5	25.7	25.7
4	22.6	26.5	27.8
5	23.3	26.3	22.5
6	23.1	25.8	24.6
7	23.5	25.9	
8	26.6	24.7	26.0
9	23.0	25.4	22.5
10	24.0	26.2	
Av.	23.4	26.0	24.4
Max.	26.6	26.8	27.8
Min.	22.2	24.7	22.1
R	4.4	2.1	5.7
V	1.52	0.41	4.08
S	1.23	0.64	2.02

TABLE 25

U.T.S. at 1450°C of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II*	*III*	*IV*	*V*	*VI(a)*	*VII*	*VIII*	*IX*	*X*	*XI(b)*	*XI(c)*
1	11.6	13.6	14.8	25.7	13.2	15.3	12.8	13.1	14.3	14.0	12.9	15.4
2	10.6	13.9	19.4	27.9	14.0	13.8	13.5	12.9	13.8	14.0	13.4	19.0
3	10.3	13.3	17.1	25.4	13.1	15.4	13.4	13.1	13.2	14.3	13.1	15.2
4	9.5	14.4	18.4	28.0	13.7	15.3	11.9	13.5	14.0	13.7	12.5	14.4
5	10.2	13.7	18.8	26.5	13.2	14.8	12.7	13.0	14.6	14.1	11.4	20.6
6	9.5	13.8	21.1	27.8	13.4	15.3	14.1	13.20		13.7	13.2	13.3
7	9.9	13.8	17.6	25.9	13.5	14.8	14.9	13.40		14.6	14.5	12.9
8	10.0	13.3	20.2	28.7	13.2	14.4	17.1	12.7		14.1	14.0	14.8
9	12.7*	14.0	18.1		13.1	14.3	19.1	12.8		14.6		
10	12.9*	13.0			12.9	13.8	17.3	12.8		14.6		
Av	10.2	13.7	18.4	27.0	13.3	14.8	14.7	13.0	14.0	14.2	13.1	15.7
Max.	11.6	14.4	21.1	28.7	14.0	15.4	19.1	13.5	14.6	14.6	14.5	20.6
Min.	9.5	13.0	14.8	25.4	12.9	13.8	11.9	12.7	13.2	13.7	11.4	12.9
R	2.1	1.4	6.3	3.3	1.1	1.6	7.2	0.8	1.4	0.9	3.1	7.7
V	0.46	0.16	3.38	1.37	0.10	0.39	5.65	0.06	0.28	0.12	0.87	7.33
S	0.68	0.4	1.83	1.17	0.32	0.625	2.37	0.26	0.53	0.34	0.93	2.71

(*a*) Longitudinal

(*b*) Transverse, sheet No.7

(*c*) Longitudinal sheet No.8

(*) Increase in strain rate after 25% strain

TABLE 26

0.2% Y.S. at 1450°C of TZM Molybdenum Alloy Sheet

Test No.	I	II	III	IV	V	VI(a)	VII	VIII	IX	X	XI
1	-	8.0	10.9	21.3	7.4	9.63	7.5	9.6	9.7	8.8	8.8
2	7.8	8.1	13.1	21.9	8.0	8.65	11.8	10.2	11.0	9.5	9.0
3	7.2	7.8	12.1	20.0	7.9	8.22	9.9	11.0	10.0	10.0	8.4
4	6.2	8.4	12.7	21.5	8.0	9.63	10.2	10.8	10.0	9.6	7.6
5	7.3	8.0	12.2	21.4	7.4	8.79	9.1	10.2	12.1	9.6	6.3
6	6.8	8.0	15.0	22.2	7.6	10.12	8.0	10.8		9.2	8.5
7	7.1	7.4	12.5	21.6	7.7	9.28	9.7	10.7		10.5	9.4
8	7.1	7.7	13.5	23.7	7.6	9.28	9.3	10.1		9.8	9.1
9	7.6*	7.9	12.1		7.7	9.21	14.4	9.6		10.4	
10	7.6*	7.3			7.9	8.50	12.6	8.7		10.2	
Av.	7.1	7.9	12.7	21.7	7.7	9.14	10.2	10.2	10.5	9.9	8.4
Max.	7.8	8.4	15.0	23.7	8.0	10.12	14.4	11.0	12.1	10.5	9.4
Min.	6.2	7.3	10.9	20.0	7.4	8.22	7.5	8.7	9.6	9.2	6.3
R	1.6	1.1	4.1	3.7	0.6	1.90	6.9	2.3	2.5	1.3	2.1
V	0.23	0.10	1.29	1.07	0.05	0.34	4.49	0.50	1.02	0.17	1.00
S	0.48	0.32	1.13	1.03	0.22	0.58	2.11	0.71	1.01	0.41	1.00

(a) Longitudinal

(*) Increase in Strain rate, after 0.5% Strain.

TABLE 27

Elongation at 1450°C of TZM Molybdenum Alloy Sheet

Test No.	*I*	*II*	*III*	*IV*	*V*	*VI(a)*	*VII*	*VIII*	*IX*	*X*	*XI(b)*	*XI(c)*
1	29.1	41.8	38.0	5.0	39.0	35.0	33.0	41.7	48.8	45.8	-	23.0
2	35.4	41.2	28.0	5.0	32.0	43.0	33.0	39.4	48.2	46.3	43.8	26.0
3	43.7	39.4	34.0	7.5	41.0	38.0	35.0	45.0	44.0	53.3	46.6	28.0
4	43.9	42.4	22.0	5.5	38.0	37.0	26.0	40.2	54.0	52.0	52.4	21.0
5	33.1	38.8	32.0	5.5	37.0	45.0	35.0	39.4	44.0	48.7	48.6	23.0
6	43.3	39.0	22.0	6.0	39.0	27.0	37.0	35.4		51.4	39.9	19.0
7	41.7	38.8	28.0	8.0	46.0	39.0	30.0	39.4		44.1	50.4	24.0
8	50.7	45.6	26.0	5.0	42.0	38.0	27.0	35.4		45.9	44.3	29.0
9	47.6*	41.9	32.0		36.0	40.0	18.0	43.3		44.2		
10	54.7*	43.0			44.0	43.0	20.0	41.4		50.0		
Av.	40.1	41.2	29.1	5.9	39.4	38.0	29.4	40.1	47.8	48.2	46.6	24.0
Max.	50.7	45.6	38.0	8.0	46.0	45.0	37.0	45.0	54.0	53.3	52.4	29.0
Min.	29.1	38.8	22.0	5.0	32.0	27.0	18.0	35.4	44.0	44.1	39.9	19.0
R	21.6	6.8	16.0	3.0	14.0	18.0	19.0	9.6	10.0	9.2	12.5	10.0
V	49.2	4.49	29.11	1.39	16.48	26.1	42.48	9.34	17.12	11.29	18.34	11.6
S	7.0	2.22	5.39	1.18	4.06	5.1	6.51	3.05	4.13	3.36	4.28	3.4

(*a*) Longitudinal

(*b*) Transverse, sheet No.7

(*c*) Longitudinal, sheet No.8

(*) Increase in Strain rate, after 0.5% Strain.

TABLE 28

Elastic Modulus at 1450°C TZM Molybdenum Alloy Sheet
1000 kg/mm²

Test No.	IV	X	XI
1	16.4	17.2	15.9
2	16.2	18.8	17.6
3	16.4	19.2	18.0
4	16.5	17.4	18.6
5	17.5	19.4	19.4
6	16.4	18.8	15.9
7	16.0	19.2	15.2
8	16.9	17.8	17.5
9		18.9	
10		19.2	
Av.	16.5	18.6	17.3
Max.	17.5	19.4	19.4
Min.	16.0	17.2	15.2
R	1.5	2.2	4.2
V	0.22	0.65	2.14
S	0.47	0.81	1.46

TABLE 29

U.T.S. at 1800°C of TZM Molybdenum Alloy Sheet

Test No.	II	III	VI(a)	VIII	IX	X	XI(b)	XI(c)
1	4.8	4.7	3.37	6.60	3.7	3.3	6.5	3.1
2	4.6	5.4	4.36	6.40	5.4	3.3	4.1	5.1
3	4.8	5.7	4.92	6.75	5.6	4.0	4.2	5.3
4	4.5	5.1	4.50	6.90	4.6	3.4	4.5	6.3
5	4.8	4.8	4.43	6.55	5.4	3.9	4.1	5.5
6	5.3	5.4	-	6.25		3.8	4.2	
7	5.4	5.3	4.99	6.20		4.0		
8	5.5	5.2	4.78	6.75		3.8		
9	5.3	4.8	4.64	6.20		3.9		
10	5.5	4.7		6.80		3.9		
Av.	5.1	5.1	4.50	6.5	4.9	3.7	4.6	5.1
Max.	5.5	5.7	4.99	6.9	5.6	4.0	6.5	6.3
Min.	4.5	4.7	3.37	6.2	3.7	3.3	4.1	3.1
R	1.0	1.0	1.62	0.7	1.9	0.7	2.4	3.2
V	0.14	0.12	0.26	0.07	0.62	0.08	0.88	1.41
S	0.38	0.34	0.51	0.27	0.79	0.28	0.94	1.19

(a) (b) (c): See note Table 27.

TABLE 30

0.2% Y.S. at 1800°C of TZM Molybdenum Alloy Sheet

Test No.	*II*	*III*	*VI(a)*	*VIII*	*XI*	*X*	*XI*
1	2.0	2.7	1.76	5.9	3.2	2.3	2.6
2	2.1	3.1	2.39	5.6	3.3	1.9	2.7
3	2.2	3.3	2.81	5.9	3.4	2.3	2.0
4	2.1	2.8	2.53	5.9	3.2	1.8	1.9
5	2.2	2.8	-	5.6	3.3	2.4	1.8
6	2.8	3.2	2.67	5.4		2.2	2.0
7	2.7	3.1	2.95	5.1		2.2	
8	2.9	2.9	2.60	5.9		2.0	
9	2.6	2.7	2.53	5.5		2.1	
10	3.0	2.7		5.6		1.8	
Av.	2.5	2.9	2.53	5.6	3.3	2.1	2.2
Max.	3.0	3.3	2.95	5.9	3.4	2.4	2.7
Min.	2.0	2.7	1.76	5.1	3.2	1.8	1.8
R	1.0	0.6	1.19	0.8	0.2	0.6	0.9
V	0.14	0.05	0.13	0.07	0.0075	0.04	0.14
S	0.37	0.22	0.36	0.26	0.087	0.21	0.38

(*a*) Longitudinal.

TABLE 31

Elongation at 1800°C of TZM Molybdenum Alloy Sheet

Test No.	*II*	*III*	*VI(a)*	*VIII*	*IX*	*X*	*XI(b)*	*XI(c)*
1	50.4	70.0	66.0	68.8	55.5	94.0	65.0	20.0
2	94.4	78.0	65.0	70.8	70.0	95.7	41.0	30.0
3	48.8	74.0	68.0	67.0	75.7	110.2	75.0	35.0
4	52.0	70.0	77.0	73.7	77.8	92.3	86.0	40.0
5	52.0	74.0	64.0	64.2	70.8	117.2	62.0	37.0
6	62.8	74.0	-	70.8		109.0		
7	49.6	72.0	73.0	73.4		101.5		
8	100.0	-	60.0	65.0		102.0		
9	52.0	80.0	75.0	67.6		109.0		
10	48.0	72.0		78.7		89.1		
Av.	61.0	73.8	68.0	70.0	70.0	102.0	65.8	32.4
Max.	100.0	80.0	77.0	78.7	77.8	117.2	86.0	40.0
Min.	48.0	70.0	60.0	64.2	55.5	89.1	41.0	20.0
R	52.0	10.0	17.0	14.5	22.3	28.1	45.0	20.0
V	382.5	11.44	35.0	19.67	76.0	84.52	280.70	61.3
S	19.55	3.38	5.92	4.43	8.72	9.19	16.75	7.84

(*a*) Longitudinal
(*b*) Transverse, sheet No.7
(*c*) Longitudinal, sheet No.8.

TABLE 32

Elastic Modulus at 1800°C of TZM Molybdenum Alloy Sheet
1000 kg/mm^2

Test No.	X	XI
1	7.80	7.9
2	7.87	11.2
3	7.50	9.6
4	7.43	9.1
5	7.90	7.0
6	8.00	
7	7.90	
8	8.33	
9	8.30	
10	7.78	
Av.	7.88	9.0
Max.	8.33	11.2
Min.	7.43	7.0
R	0.90	4.2
V	0.08	2.60
S	0.29	1.61

TABLE 33

Tensile Properties of Vascojet 90 Sheet at Room Temperature: Conventional Conditions
UTS and YS: kg/mm^2; El.:%; Mod.: 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
I	10	62.64	4.3	49.02	4.6	20.0	3.9	-	-
II	10	64.03	1.9	50.37	3.7	19.7	5.3	20.3	4.60
III	10	66.24	2.0	50.64	2.1	18.6	5.0	-	-
V	10	64.91	1.4	50.45	1.7	20.7	3.0	-	-
VI	10	67.12	2.4	50.65	1.9	17.1	4.0	-	-
VII	10	64.85	2.8	50.4	1.8	20.6	3.0	-	-
VIII	10	63.46	1.0	50.3	1.8	18.3	3.9	-	-
X	10	64.86	1.3	50.89	1.2	18.9	3.8	20.93	1.90
XI	10	63.85	2.3	49.16	1.5	17.7	3.6	21.52	2.90
		64.56	2.17	50.2	2.25	19.1	3.94	20.91	3.13

TABLE 34

Tensile Properties of Vascojet Steel at Room Temperature: High Temperature Conditions
UTS and YS; kg/mm^2; El. : %, Mod. : 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
I	10	64.44	2.10	48.62	1.20	19.3	2.0	-	-
II	10	64.04	1.60	50.11	2.40	20.1	6.3	-	-
III	10	65.27	4.10	49.77	3.30	18.9	5.0	-	-
IV	10	69.09	1.40	53.68	1.40	18.7	4.0	20.87	1.10
V	10	65.91	1.20	50.23	2.40	19.1	3.0	-	-
VI	10	67.93	2.70	51.28	2.90	16.7	3.0	-	-
VII	10	64.10	2.20	49.49	3.40	20.0	2.0	-	-
VIII	10	62.81	1.70	48.95	2.80	17.9	3.9	-	-
X	10	64.20	2.50	50.87	1.80	18.0	3.1	20.94	1.10
XI	10	65.95	5.00	50.98	5.50	17.1	4.8	22.24	5.80
		65.33	2.44	50.40	2.71	18.4	3.7	21.32	2.66

TABLE 35

Effect of the Strain Measurement and Strain Rate Method on the Average 0.2% Yield Strength of Vascojet and TZM.

Material	Laboratories	No. of Tests	Crosshead Av.	Crosshead S	Extensometer Av.	Extensometer S
Vasc. conv.	II, V, VI, VII, X, XI,	60	-	-	50.3	0.83
Vasc. conv.	I, III, VIII	30	50.0	1.32	-	-
Vasc. HT cond.	IV, VII, X, XI,	39	-	-	51.30	1.84
Vasc. HT cond.	I, II, III, V, VI, VIII	59	49.8	1.15	-	-
TZM RT	IIa, IIb, V, VII, IX, X, XI	72	-	-	76.5	3.63
TZM RT	I, VIII	20	77.0	3.97	-	-
TZM 1050	IV, IX, X, XI,	34	-	-	48.3	2.08
TZM 1050	I, II, III, V, VII, VIII	53	47.7	4.29	-	-
TZM 1450	IX, X, XI,	23	-	-	9.50	1.15
TZM 1450	I, II, III, V, VIII	46	9.15	2.15	-	-
TZM 1800°C	IX, X, XI,	21	-	-	2.4	0.56
TZM 1800°C	II, III, VIII	30	3.68	1.45	-	-
Material	Laboratory	No. of Tests	Constant Crosshead speed Av.	Constant Crosshead speed S	Constant Strain Rate Av.	Constant Strain Rate S
Vasc. Conv.	II, V, X	30	-	-	50.6	0.76
Vasc. Conv.	I, III, VI, VII, VIII, X	60	50.0	1.10	-	-
Vasc. HT cond.	IV, X,	20	-	-	52.3	1.52
Vasc. HT cond.	I, II, III, V, VI, VII, VIII, XI	78	49.9	1.25	-	-
TZM RT	II, IV, X, XI	40	-	-	77.2	4.44
TZM RT	I, V, VII, VIII, IX	44	76.2	2.92	-	-

TABLE 36

Tensile Properties of TZM sheet (Transverse) at Room Temperature
UTS and YS: kg/mm^2; El. : %; Mod. 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
I	10	83.4	5.3	73.6	3.3	15.4	5.0	-	-
IIa	10	87.4	2.8	76.1	2.3	19.6	4.8	30.34	11.30
IIb	8	89.5	5.6	75.9	9.6	18.1	5.9	-	-
IV	11	91.5	8.4	84.2	5.8			30.1	2.0
V	10	89.9	4.1	75.2	3.5	18.2	4.0	-	-
VII	10	89.0	4.9	75.6	6.4	18.7	5.0	-	-
VIII	10	91.2	12.6	80.4	8.5	18.2	4.3	-	-
IX	4	91.9	4.0	76.4	1.0	17.5	8.0	-	-
X	10	90.4	3.5	75.7	1.8	16.1	7.9	20.6	1.6
XI	10	85.7	7.8	72.9	6.6	15.1	8.8	29.6	4.2
Av.		88.8	5.9	76.6	4.8	17.4	6.0	30.1	4.8

TABLE 37

Tensile Properties of TZM sheet (Longitudinal) at Room Temperature

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
VI	10	92.1	2.1	80.8	1.4	9.0	10.0	25.2	2.7
XI	10	89.6	8.6	83.0	7.9	11.4	4.1	30.2	3.6
Average		90.8	5.3	81.9	4.6	10.2	7.0	27.7	3.1

TABLE 38

Tensile Properties of TZM Sheet (Transverse) at 1050°C
UTS and YS: kg/mm^2; El. : %; Mod. : 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
I	8	54.0	3.5	43.5	6.3	7.2	2.7	-	-
II	10	54.0	2.2	43.1	9.2	7.1	3.3	-	-
III	6	54.7	1.4	46.3	7.7	6.3	2.0	-	-
IV	10	52.9	4.4	49.1	2.9			23.4	4.4
V	10	55.1	1.4	47.3	2.2	6.3	2.0	-	-
VII	11	59.7	5.6	53.2	5.2	6.2	3.0	-	-
VIII	10	56.1	5.9	50.2	6.8	6.6	4.0	-	-
IX	5	59.8	2.7	44.5	1.3	5.1	2.6	-	-
X	10	56.5	2.0	49.8	2.4	5.9	1.2	26.0	2.1
XI	9	54.8	12.3	48.0	6.0	5.5	3.6	24.4	5.7
Av.		55.7	4.1	48.0	5.0	6.3	2.7	24.6	4.0

TABLE 39

Tensile Properties of TZM Sheet (Longitudinal) at 1050°C

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range
VI	10	59.8	4.4	51.12	9.5	5.0	5.0
XI	7	46.0	12.4	-	-	5.6	6.1
Av.		54.1	8.4			5.2	5.5

TABLE 40

Tensile Properties of TZM Sheet (Transverse) at 1450°C
UTS and YS: kg/mm^2; El. : %, Mod. : 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
I	8	10.2	2.1	7.1	1.6	40.1	21.6	-	-
II	10	13.7	1.4	7.9	1.1	41.2	6.8	-	-
III	9	18.4	6.3	12.7	4.1	29.1	16.0		
IV	8	27.0*	3.3*	21.7*	3.7*	5.9*	3.0*	16.5	1.5
V	10	13.3	1.1	7.7	0.6	39.0	14.0	-	-
VII	10	14.7	7.2	10.2	6.9	29.4	19.0	-	-
VIII	10	13.1	0.8	10.2	2.3	40.1	9.6	-	-
IX	5	14.0	1.4	10.5	2.5	47.8	10.0	-	-
X	10	14.2	0.9	9.9	1.3	48.2	9.2	18.6	2.2
XI	8	13.1	3.1	8.4	3.1	46.6	12.5	17.3	4.2
Av.		13.9	2.7	9.4	2.7	39.6	13.2	18.0	3.2

* These results were ommitted in the Calculation (see Text).

TABLE 41

Tensile Properties of TZM Sheet (Longitudinal) at 1450°C

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range
VI	10	14.8	1.6	9.1	1.90	38.0	18.0
XI	8	15.7	7.7	-	-	24.0	10.0
Av.		15.2	4.6			32.0	14.0

TABLE 42

Tensile Properties of TZM Sheet((Transverse) at 1800°C
UTS and YS: kg/mm^2: El : %, Mod. : 1000 kg/mm^2

Laboratory	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range	Modulus Average	Modulus Range
II	10	5.1	1.0	2.5	1.0	61.0	52.0	-	-
III	10	5.1	1.0	2.9	0.6	74.0	10.0	-	-
VIII	10	6.5	0.7	5.6	0.8	70.0	14.5	-	-
IX	5	4.9	1.9	3.3	0.2	70.0	22.3		
X	10	3.7	0.7	2.1	0.6	102.0	28.1	7.88	0.90
XI	6	4.6	2.4	2.2	0.9	65.8	45.0	8.96	4.20
Av.		5.0	1.3	3.1	0.7	74.9	28.7	8.24	2.55

TABLE 43

Tensile Properties of TZM Sheet (Longitudinal) at 1800°C

Laboratories	Number of Tests	UTS Average	UTS Range	YS Average	YS Range	Elongation Average	Elongation Range
VI	9	4.5.	1.62	2.53	1.19	68.0	17.0
XI	5	5.1	3.2			32.4	20.0
Av.		4.7	2.4			55.2	18.5

TABLE 44

Contamination of TZM Sheet (Laboratory VIII)

Condition	Sample	O_2 ppm	N_2 ppm	H_2 ppm	C ppm
Delivery	Massive	< 10	< 30	1 max.	295
	Chips	30	50	1 max.	318
Tested at RT	Massive	< 10	< 30	1 max.	290
	Chips	35	50	1 max.	295
Tested at 1050°C	Massive	20	< 30	1 max.	315
	Chips	120	70	1 max.	280
Tested at 1450°C	Massive	25	< 30	1 max.	323
	Chips	100	80	1 max.	318
Tested at 1800°C	Massive	25	< 30	1 max.	305
	Chips	90	65	1 max.	295

TABLE 45

Properties of Vascojet 90 Steel Sheet
UTS and 0.2% YS: kg/mm^2; El : %; Mod., 10^3 kg/mm^2

Property		No. of Labs.	No. of Tests	Average	Std. Deviation	%
Conv.	UTS	9	90	64.6	1.58	2.5
Conv.	0.2% YS	9	90	50.2	1.02	2.02
Conv.	El.	9	90	19.1	1.69	8.8
Conv.	Mod.	3	30	20.91	1.07	5.1
HT	UTS	10	100	65.3	1.99	3.0
HT	0.2% YS	10	98	50.4	1.61	3.2
HT	El.	10	99	18.4	1.65	9.0
HT	Mod.	3	29	21.32	1.18	5.5

TABLE 46

Transverse Properties of TZM Molybdenum Alloy Sheet
UTS and 0.2% YS: kg/mm^2; El : %; Mod. : $10^3 kg/mm^2$

Property		No. of Labs.	No. of Tests	Average	Std. Deviation	%
RT	UTS	10	93	88.8	3.33	3.8
RT	0.2 YS	10	92	76.6	3.69	4.8
RT	El	9	81	17.4	2.44	14.0
RT	Mod.	4	40	30.13	2.14	7.1
1050°C	UTS	10	89	55.7	2.68	4.8
1050°C	0.2 YS	10	87	48.0	3.58	7.5
1050°C	El	9	79	6.3	1.13	18.0
1050°C	Mod.	3	28	24.62	1.71	7.0
1450°C	UTS	9	80	13.9	2.26	16.3
1450°C	0.2 YS	8	69	9.4	1.91	20.3
1450°C	El	9	79	39.6	7.99	20.2
1450°C	Mod.	2	18	18.0	1.30	7.2
1800°C	UTS	6	51	5.03	1.01	20.1
1800°C	0.2 YS	6	51	3.15	1.32	42.0
1800°C	El	6	49	75.0	18.2	24.2
1800°C	Mod.	2	15	8.24	1.03	12.5

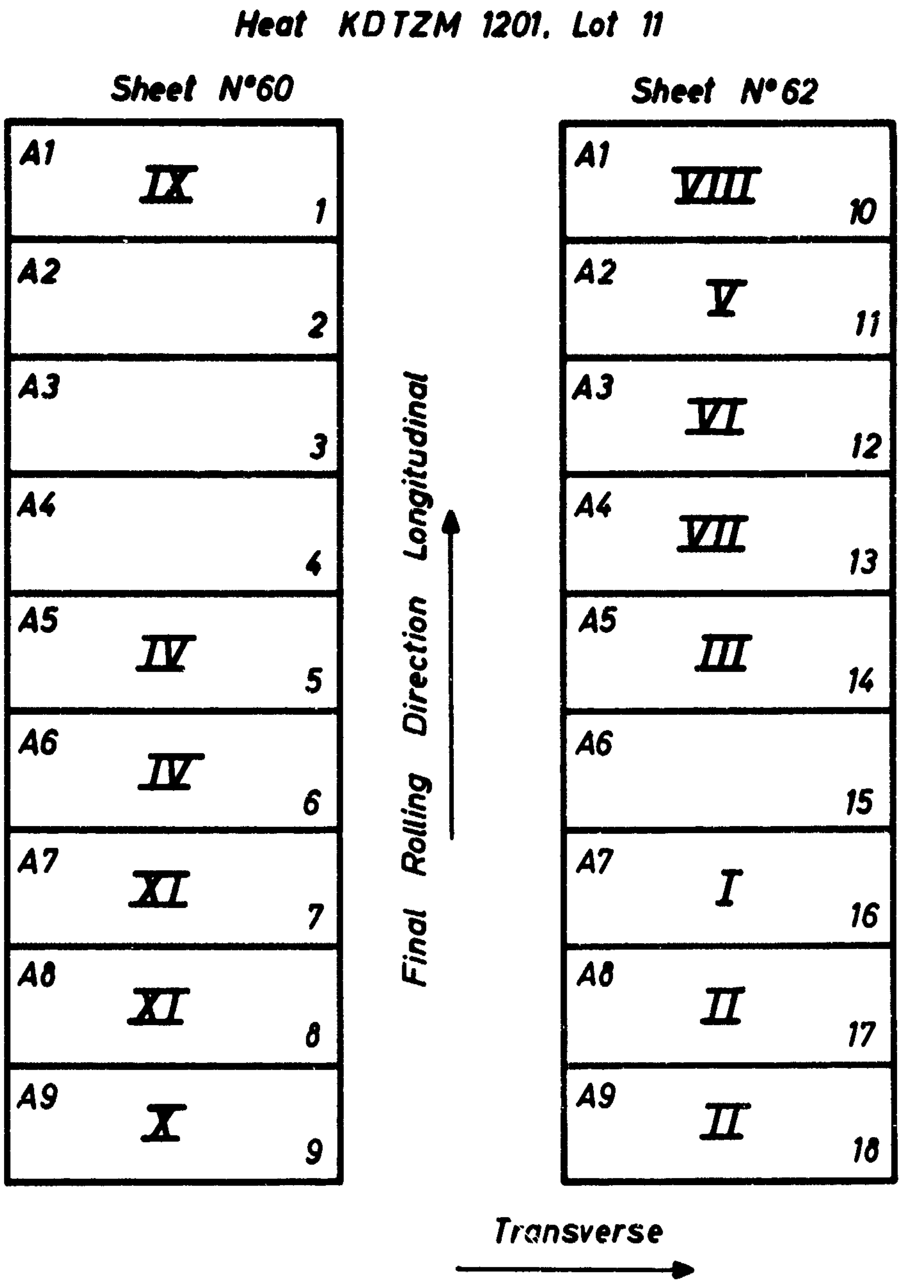


Fig. 1 Distribution of the TZM sheet material

1. I	2. X	3. IX
4. II	5. III	6. XI
7.	8. IV	9. VII
10. VI	11. VIII	12. V

Fig. 2 Distribution of the Vascojet 90 steel sheets

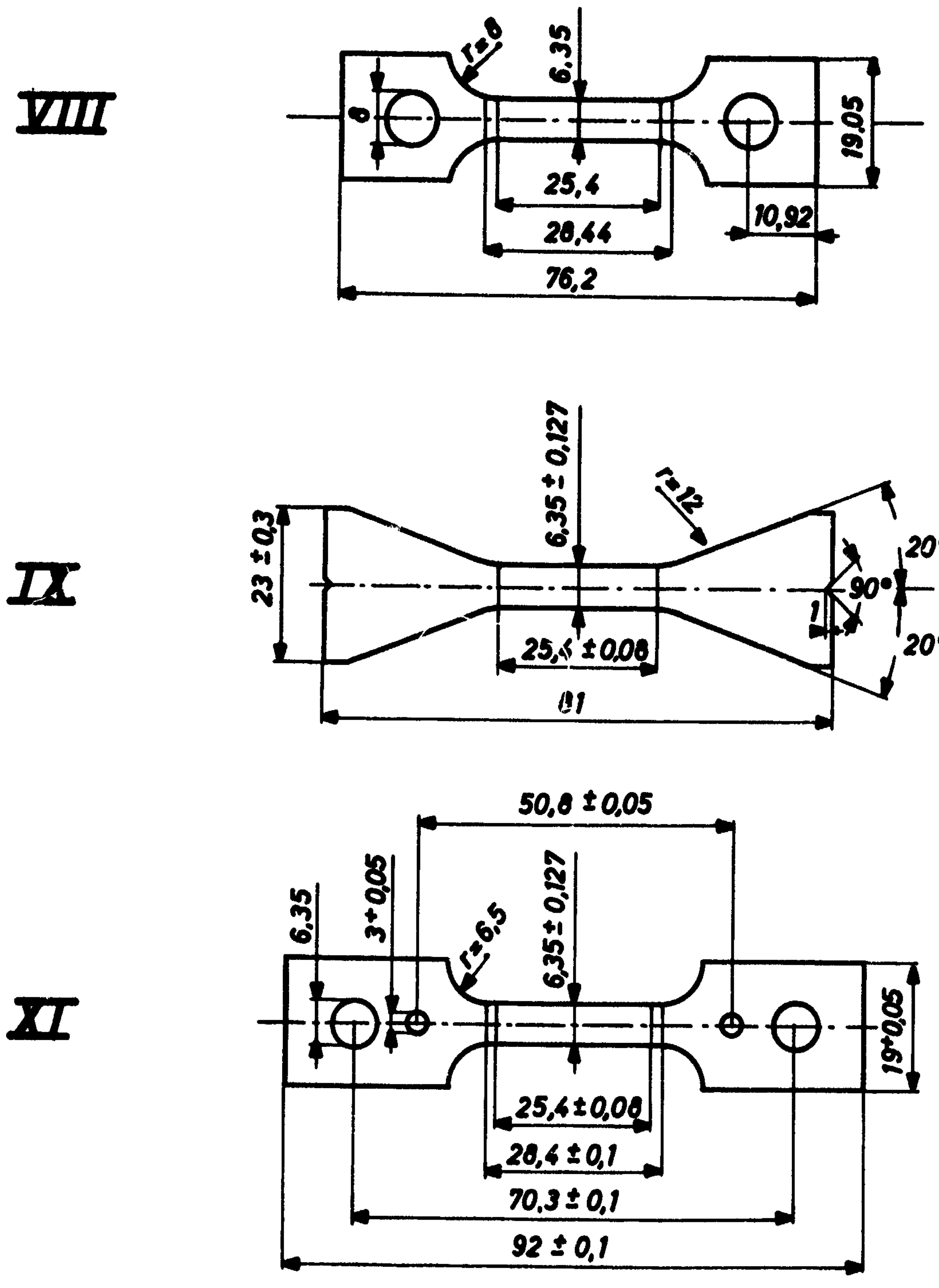


Fig.3a Test pieces used by laboratories VIII, IX and XI

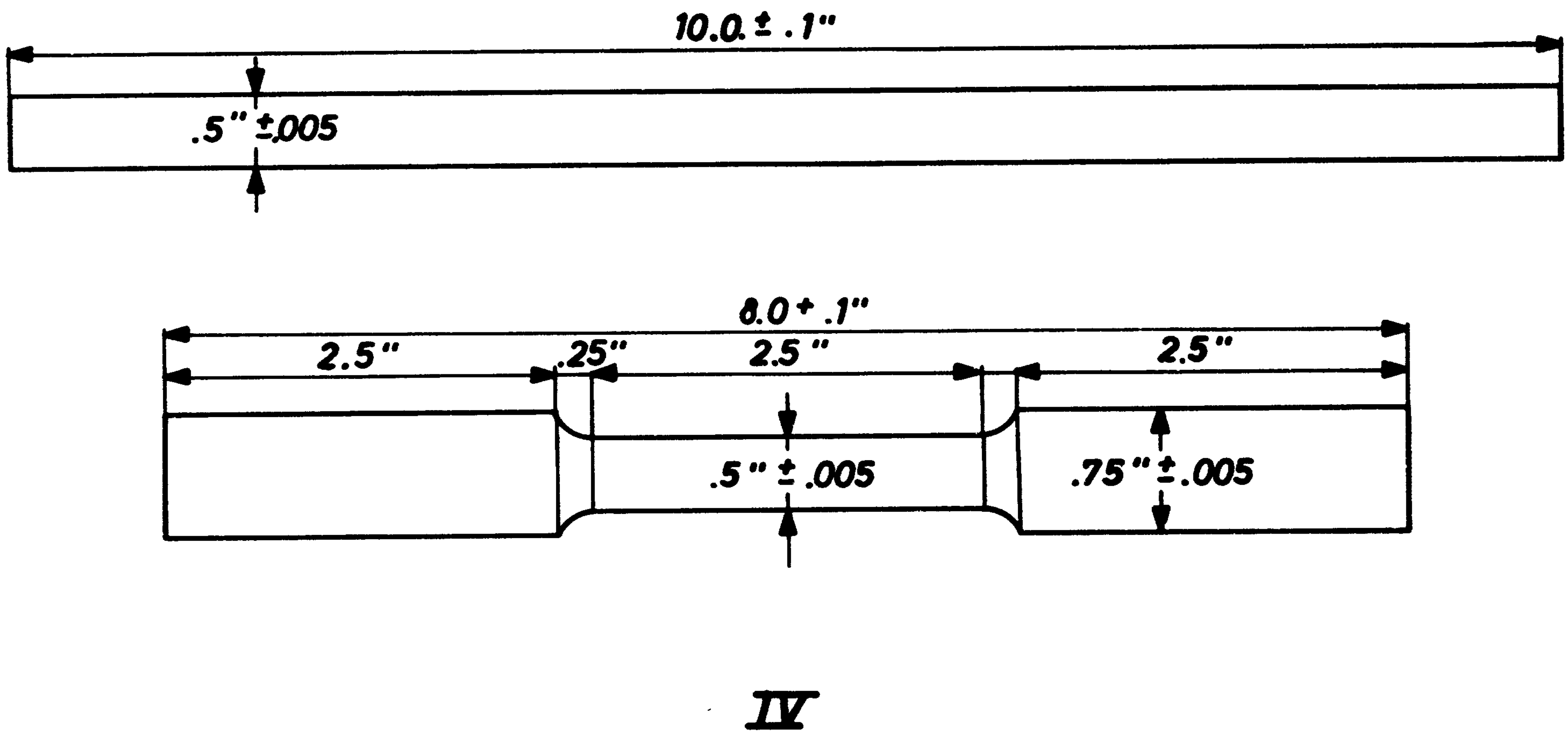


Fig. 3b Test pieces used by laboratory IV

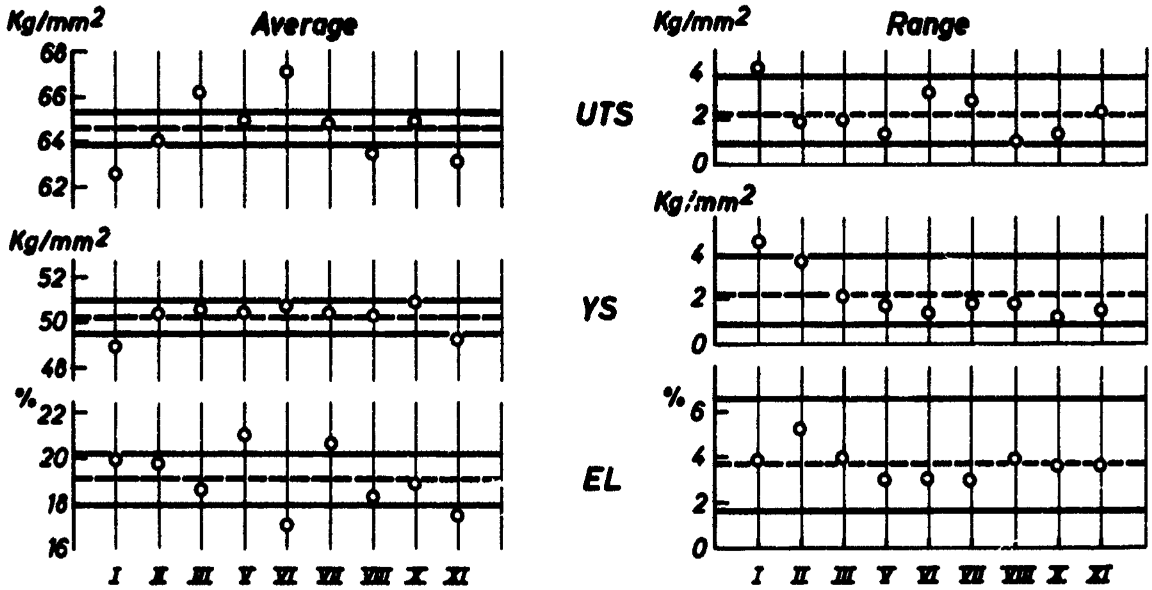


Fig.4 Conventional tests on Vascojet 90 steel sheet

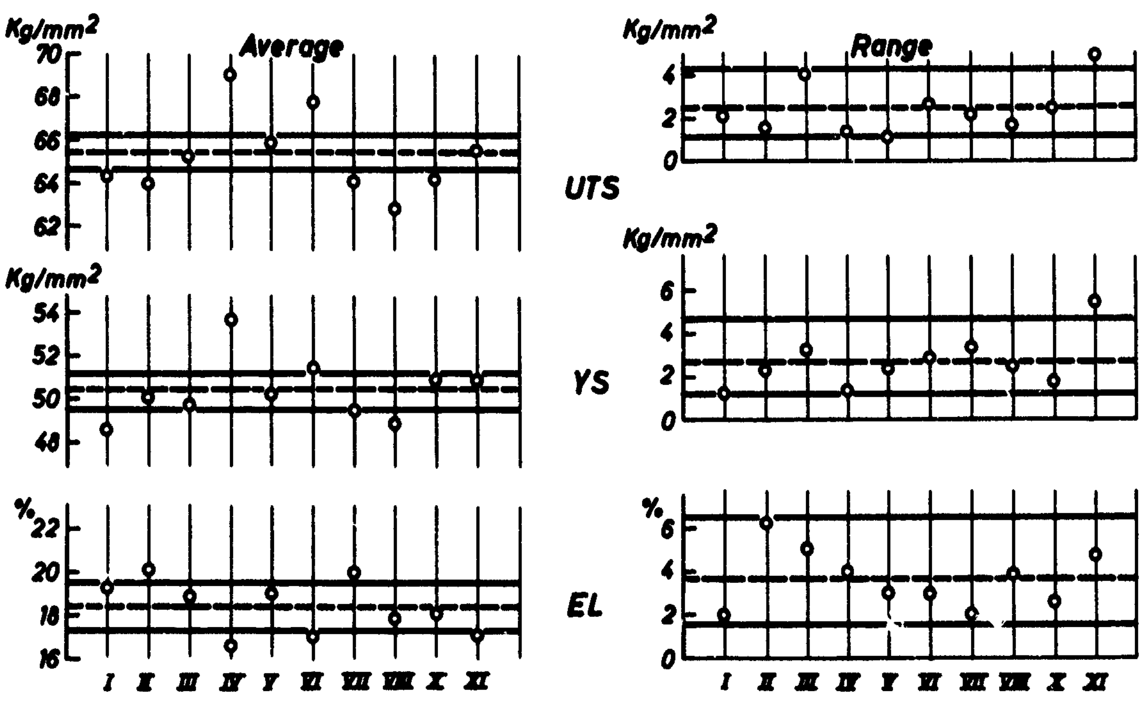


Fig.5 Tests on Vascojet 90 steel sheet under high temperature conditions

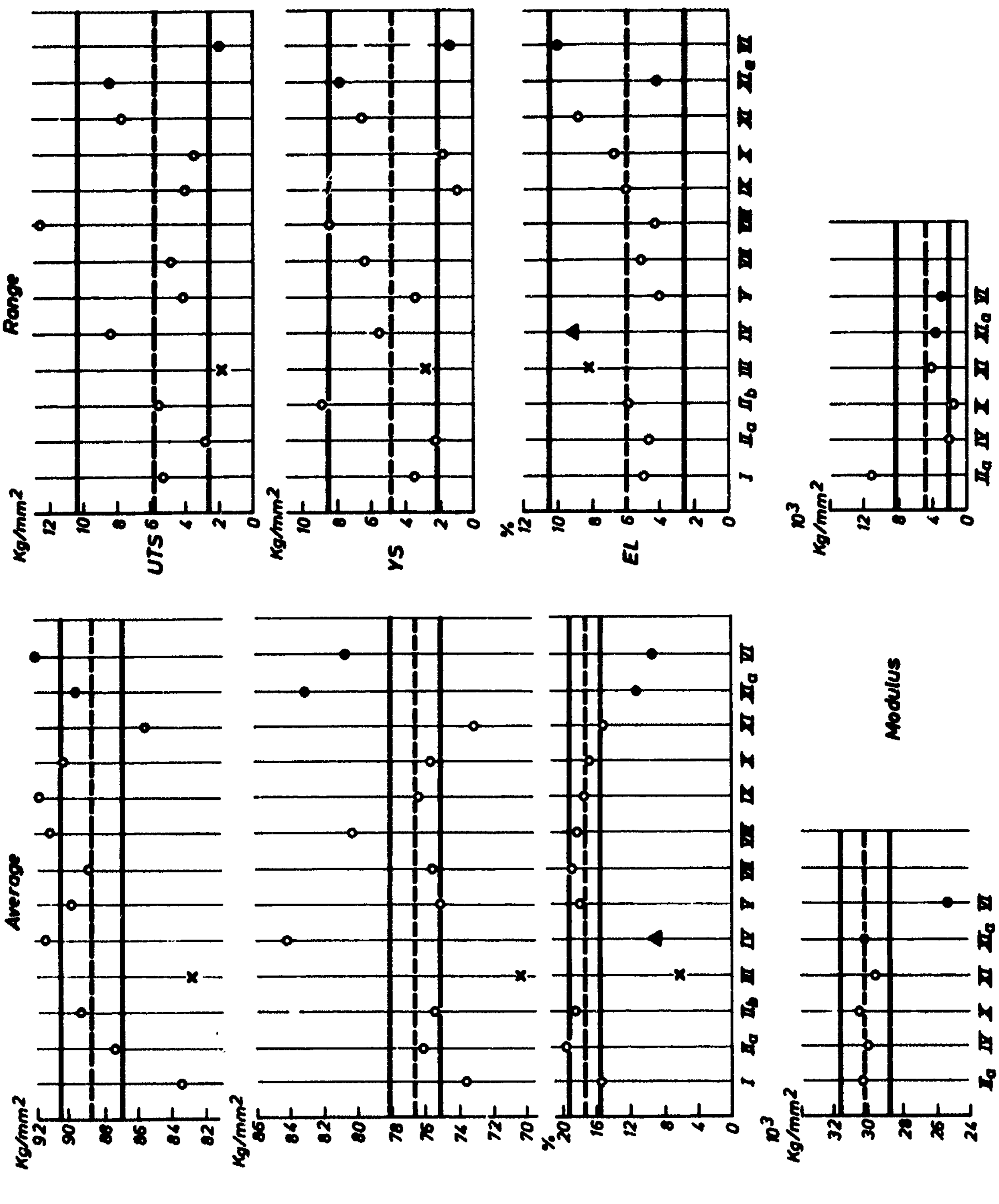


Fig. 6 Tensile tests on TZM sheet at R.T. only the transverse properties were used for the calculation of averages and limits

o : transverse o : longitudinal x : only the results ▲: 2 inches gauge length

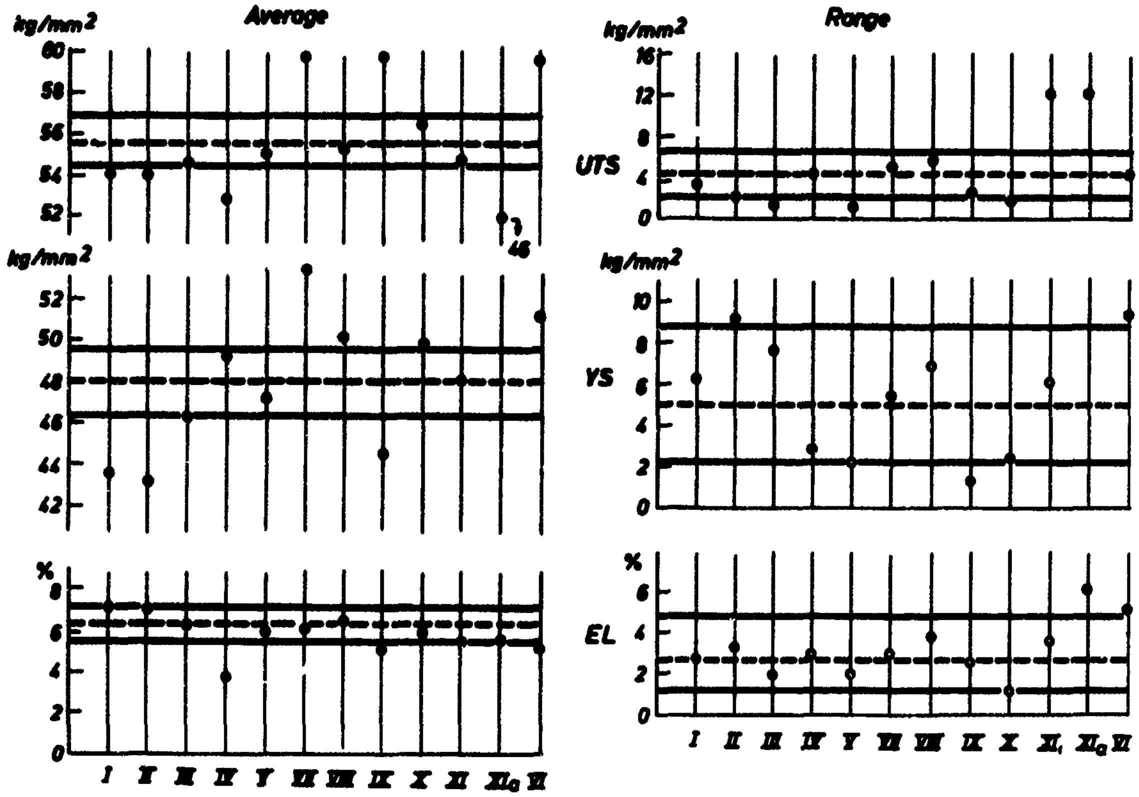


Fig.7 Tensile tests on TZM sheet at 1050°C. o : transverse •: longitudinal. Only transverse properties were used for the calculation of averages and limits

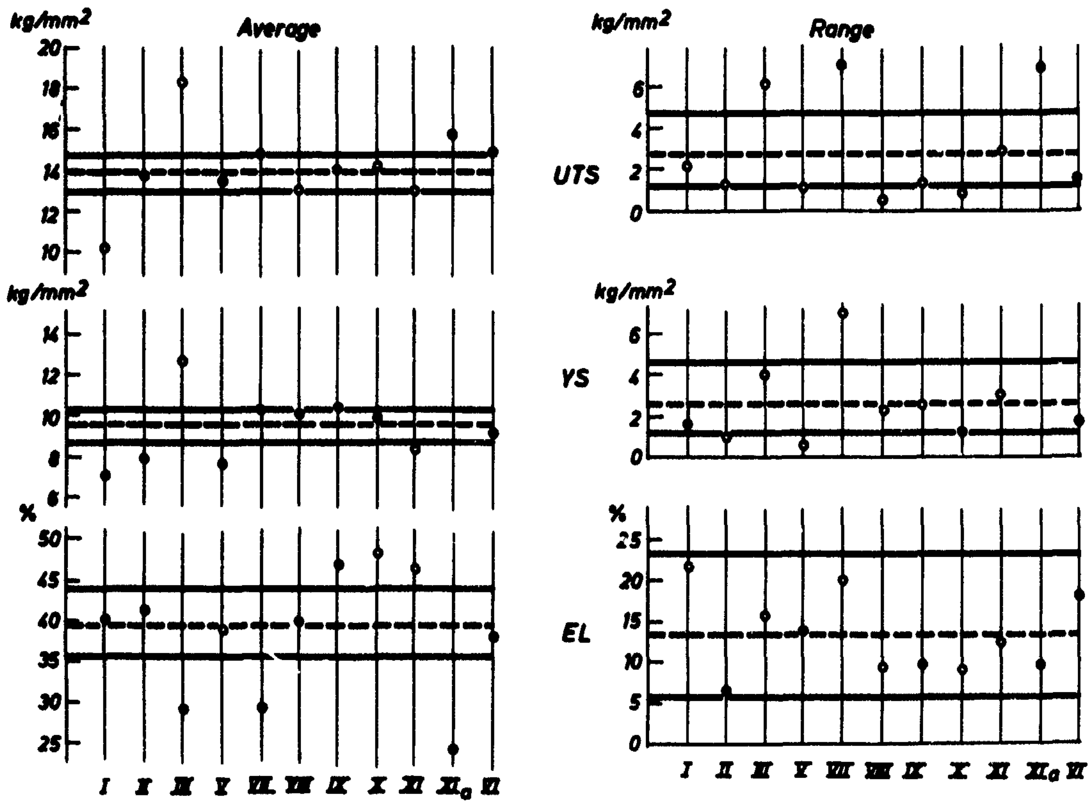


Fig.8 Tensile tests on TZM sheet at 1450°C. o : transverse •: longitudinal. Only transverse properties were used for the calculation of averages and limits

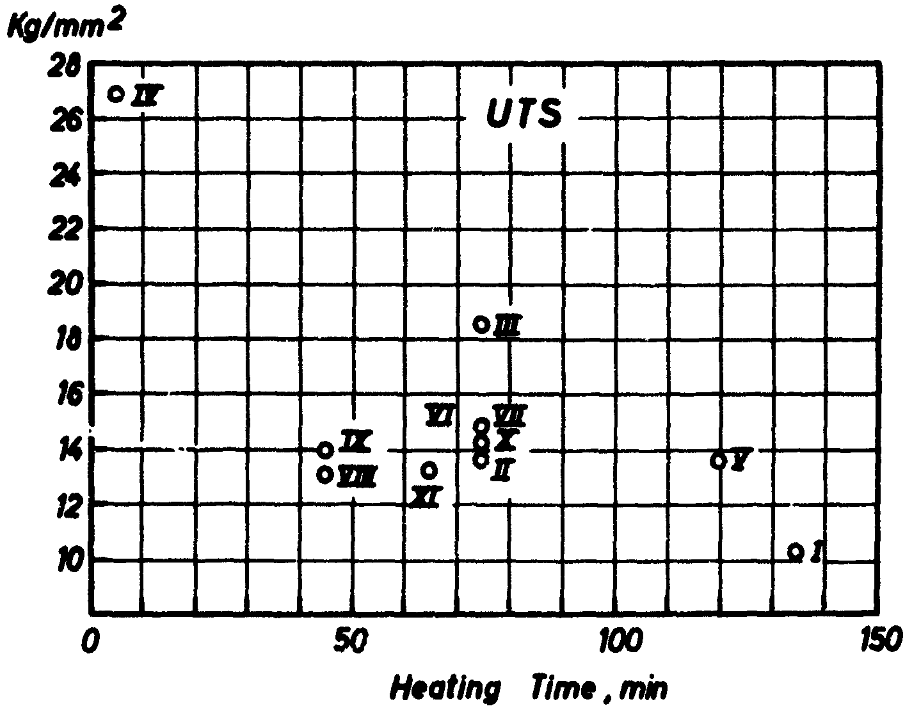


Fig.9 UTS of TZM sheet at 1450°C as a function of heating + holding time at temperature

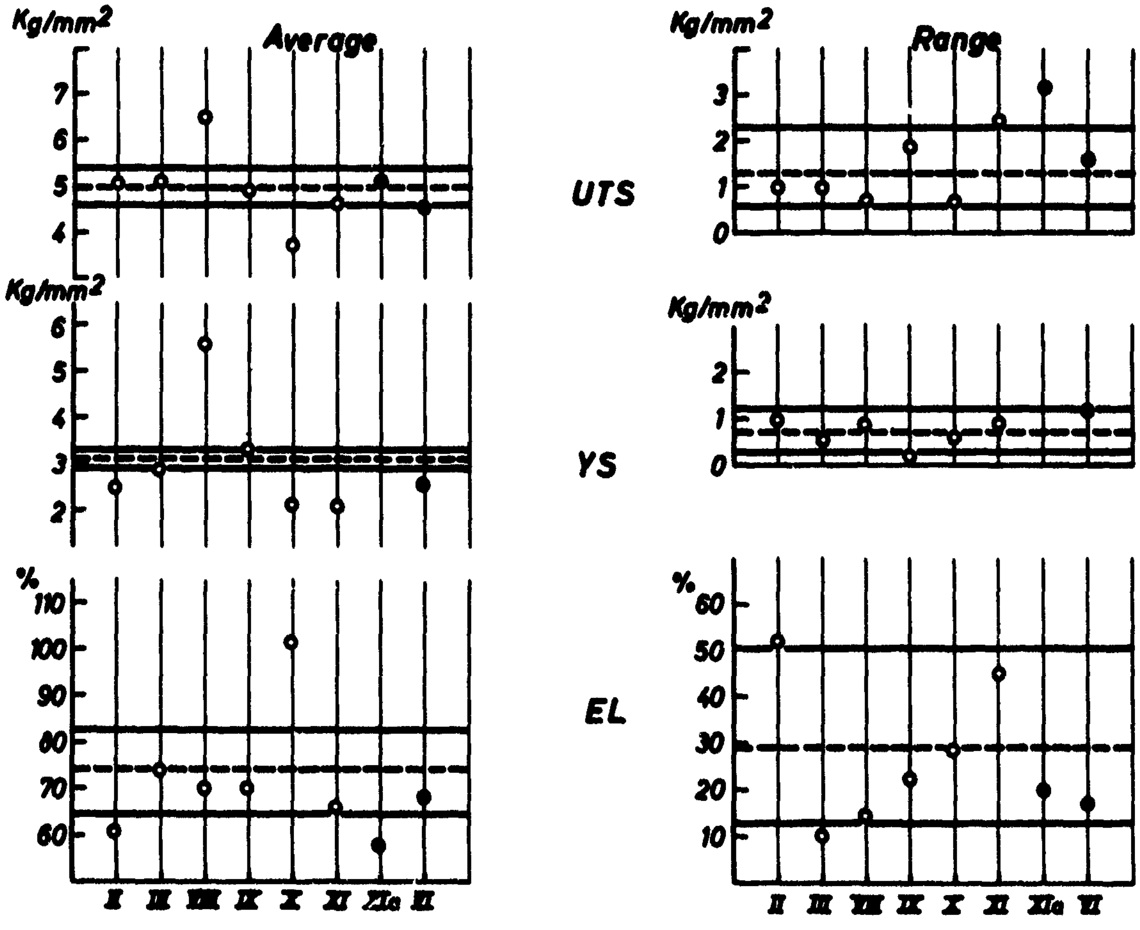


Fig.10 Tensile tests on TZM sheet at 1800°C. o : transverse ● : longitudinal

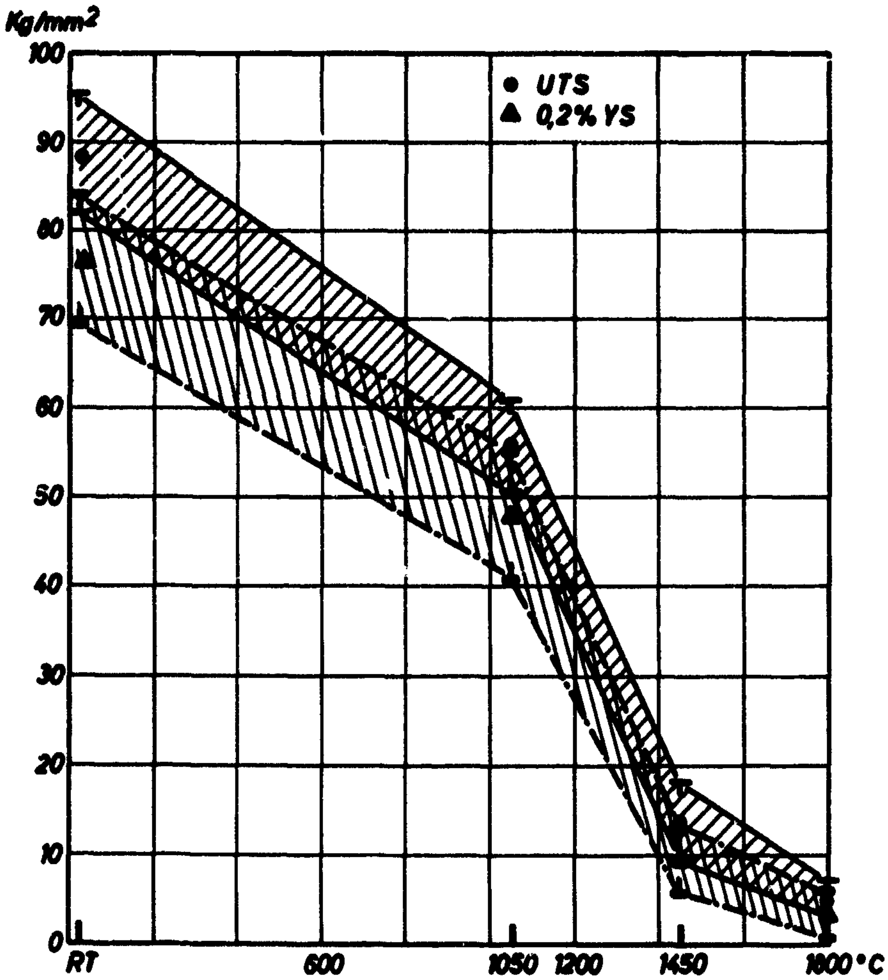


Fig. 11 0.2% YS and UTS as a function of temperature and their 95% confidence limits for the transverse direction (table 45)

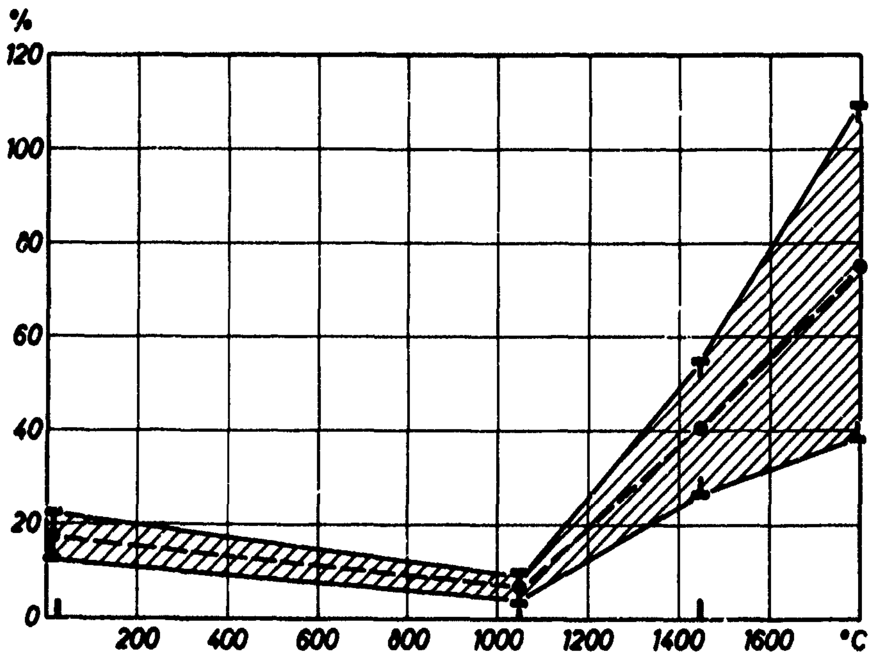


Fig. 12 Elongation as a function of temperature and its 95% confidence limits for the transverse direction of TZM sheet

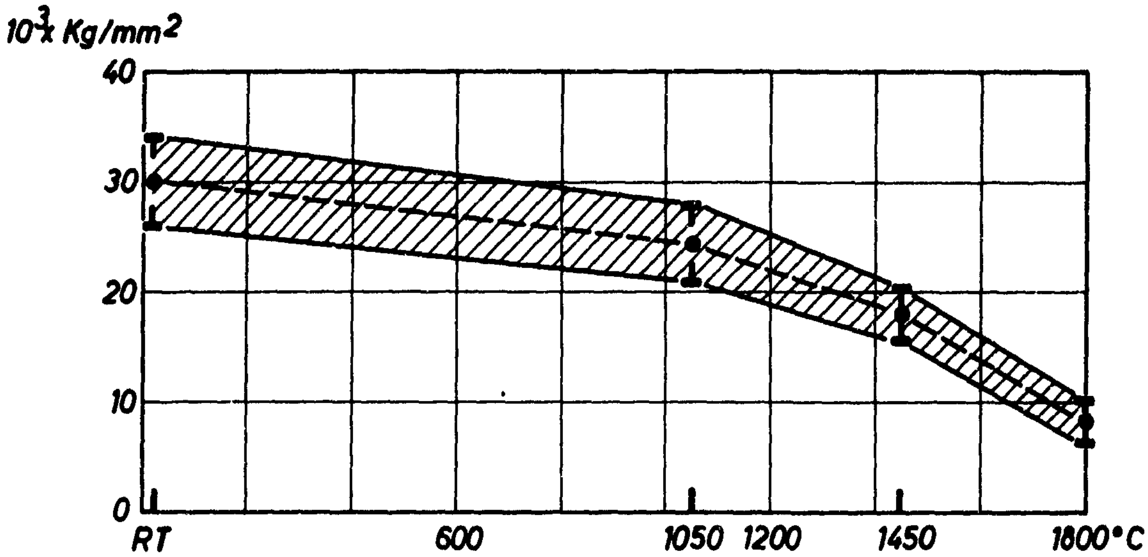


Fig. 13 Elastic Modulus as a function of temperature and its 95% confidence limits for the transverse direction of TZM sheet

APPENDIX A

Document Used for the Cooperative Programme on TZM Molybdenum Alloy Sheet

1. SCOPE OF THE PROGRAMME

The purpose of the present Round Robin Programme on mechanical properties evaluation of refractory metals is:

(a) to verify the adequacy of the specifications relative to the tests that shall be performed and to provide data for the eventual improvement of these specifications.

(b) to determine the relative precision and accuracy of the test methods that will be used in the participating laboratories and to provide data for the eventual improvement of these methods.

2. TEST REFRACTORY METAL MATERIAL

2.1 Definition of the Material

TZM Molybdenum alloy sheet material was made available to this Round Robin Programme through the courtesy of USA. The material was produced by Universal Cyclops Steel Corporation.

2.2 Identification

According to the producer, the identification and manufacturing data of the material are as follows:

Heat: KDTZM, Lot: 11, Sheets: 60 and 62, Gauge: 0.060 in.

2.3 Manufacturing Data

According to the producer the material was processed as follows:

Melt 8 in. diam. ingot, extrude to 4.1/4 in. diam. at $\sim$ 2100°F

Anneal 1 hr at $\sim$ 2800°F, In Fab forge at $\sim$ 3400°F to 1.5 in. thick

Anneal 1 hr at $\sim$ 2800°F, roll at $\sim$ 2200°F to 0.760 in. thick

Anneal 1 hr at $\sim$ 2700°F, roll at $\sim$ 2200°F to 0.265 in. thick

Anneal 1 hr at $\sim$ 2150°F, cross roll at $\sim$ 1800°F to 0.066 in. thick

Anneal 1 hr at $\sim$ 2300°F, belt grind and pickle to 0.060 in. thick.

2.4 Chemistry

At an indication we give here below the producer's results on the composition of two sheets originating from the same lot as the material under test:

	Heat: KDTZM 1201 Lot: 11 Sheet: 57 Gauge: 0.060	Heat: KDTZM 1201 Lot: 11 Sheet: 58 Gauge: 0.060
C	0.032%	0.031%
Ti	0.51	0.55
Zr	0.11	0.083
Si	< 0.0035	< 0.0035
Fe	0.0015	< 0.0015
Ni	< 0.001	< 0.001
O_2	0.0008	0.0013
N_2	0.0003	0.0003
H_2	0.0001	0.00033

2.5 Presentation

18 sheets of 8" x 24" x 0.060" cut out of original sheets of 72" x 24" are identified as follows:

Sheets No.	*Identification*
1	Universal Cyclops TZM, lot 11, sheet 60, A1
2	" " " " " A2
3	" " " " " A3
4	" " " " " A4
5	" " " " " A5
6	" " " " " A6
7	" " " " " A7
8	" " " " " A8
9	" " " " " A9
10	" " " " sheet 62, A1
11	" " " " " A2
12	" " " " " A3
13	" " " " " A4
14	" " " " " A5
15	" " " " " A6
16	" " " " " A7
17	" " " " " A8
18	" " " " " A9

3. TESTS

The following tests will be carried out in this programme:

(a) Tensile strength test at room temperature, 1050°C, 1450°C and 1800°C. Ten (10) tests shall be conducted at each temperature on specimens machined in the longitudinal directions. The number of tests at each temperature was set up in order to obtain reproducibility date within each laboratory. The specifications for these tests are given in Appendix I.

(b) Determination of recrystallization temperature. Specifications for this type of test are given in Appendix II.

4. REPORTS

The experimental conditions and results shall be reported according to the indications given in the specifications (Appendix I and II). A copy of the report shall be forwarded to the coordinator.

Liège, September 1964.

D. COUTSOURADIS.
Agard Coordinator.

N.B. The specifications given in the Appendix are mainly based on Agard Report WP M27. Second Issue, Supplement, November 1962. Some slight modifications have been, however, performed taking into account MAB Report 192-M, April 22, 1963.

APPENDIX I to APPENDIX A

1. TENSILE STRENGTH TEST, ROOM TEMPERATURE AND ELEVATED TEMPERATURE

1.1 Test Piece Dimensions

The test pieces for sheet material shall be of the following dimensions:

	Inches	mm
Gauge length	1.000 ± 0.003	25.4 ± 0.08
Width	0.250 ± 0.005	6.35 ± 0.127
Radius of Fillet	0.250 min.	6.35 min.

The maximum permissible variation in a single uniform width specimen shall be of 0.001 inch. If desired, the width at the centre may be reduced by not more than 0.005 inch to favour a centre fracture. In this case, the width must be uniformly tapered from both ends to the centre, the end widths may not differ by more than 0.001 inch, and the entire gauge sections must be within the 0.250 ± 0.005 limits. The most important consideration is accuracy of measurement of width and thickness. An accuracy of 0.2 per cent, should be assured and sufficient measurements should be made to clearly establish the position and exact dimensions of the minimum cross section.

Typical specimens are given in Figure 1.

The recommended small size test pieces are intended particularly for preliminary evaluation of small pilot lots of new alloys. The smaller size provides several advantages:

(1) requires less material,

(2) allows more rapid heating,

(3) improves gauge temperature uniformity,

(4) reduces grip loads.

1.2. Gripping Method

Various types of gripping devices may be used to transmit the measured load applied by the testing machine to the test pieces. To insure axial tensile stress within the gauge length, the axis of the test specimen should coincide with the centre line of the heads of the testing machine. Provided that no bending stresses are introduced, any gripping device is acceptable. The gripping method used shall be described in the report.

1.3 Surface Finish

The gauge section surface finish (except for the edges) shall be the same as delivered mill finish; no additional grinding, machining, pickling, or other surface finishing is permitted. The edges of the gauge section, shall be carefully deburred and polished longitudinally with 00 emery so that all polishing scratches are parallel to the long direction edge. Previously to the polishing operation 0.025 in (0.635 mm) shall be removed by milling or filing from all sheared or punched edges.

Note 1. The recommended surface finish has particularly the purpose of insuring the same surface finish for all the test pieces to be tested in the frame of the AGARD round robin testing programme.

1.4 Pretest Inspection

The sheet material shall be examined carefully for surface contamination. This examination shall include:

- determination of microhardness normally to the surface,
- metallographic examination of the as received sheet and of recrystallized specimen (see Appendix II),
- chemical analysis for oxygen, nitrogen, hydrogen and carbon on chips representing a cut of 0.005 to 0.010 inch (0.127 to 0.254 mm), from the surface.

The analysis of this surface sample shall be compared with gross sample analysis.

Note 2. For the chemical analysis, evidence shall be given for the reliability of the sampling procedure and for the precision of the method of analysis used.

The test pieces shall be checked for cracks prior to test by low power optical inspection and fluorescent penetrant inspection. Reference shall be made to relevant standards, as used in different countries.

1.5 Loading Apparatus and Methods

The type of testing machine is not specified but shall be reported with the results of tests. Evidence for the accuracy of the machine shall be given according to specifications for calibrating tensile test machines that are available in many countries.

Precautions shall be taken to assure that the load on the test piece is applied as nearly axially as possible. These precautions shall be described.

Eccentricity of loading can often be detected by elastic extension measurements taken at room temperature. Apparatus provided with extensometers affording separate measurements on opposite sides will reveal unsatisfactory alignment in respect to one plane when unequal strain is shown by the readings on opposite sides. Repeating this procedure with the points of attachment to the test piece at 90 deg. to the first eccentricity test will help define the extent and orientation of any eccentricity. Such measurements shall be taken periodically on a gauged test piece at room temperature to check axial alignment. Extension measuring devices which show extension on one side of the test piece only do not readily lend themselves to the detection of eccentric loading. It should be noted that gripping devices tend to show some creep with repeated use at elevated temperatures and may lose their axiality. They should be periodically examined with care for determining any such nonaxiality and reworked when necessary.

For high-temperature testing of materials which are readily attacked by their environment (such as oxidation of metal in air), the sample may be enclosed in a capsule so that it can be tested in a vacuum or inert gas atmosphere. When such equipment is used, the necessary corrections to obtain true test piece loads must be made. For instance, compensation must be made for difference in pressure inside and outside of the capsule and for any load variation due to sealing ring friction, bellows or other features.

1.6 Strain Measurement and Strain Rate for Tests up to and including 850°C

1.6.1 Method

An extensometer, preferable recording, shall be used to determine strain from zero to a minimum of 0.5 per cent offset. For offset yield strength determination, the accuracy of the extensometer shall be of 0.1 per cent or better. For Young's modulus determination

the maximum error of the extensometer shall be of 0.01 per cent or less. For strain measurement beyond the 0.5 per cent offset strain, it is permissible to use gauge marks instead of extensometer measurement.

1.6.2 Strain Rate

A strain rate of 0.005 ± 0.001 per minute shall be used to 0.5 per cent offset. Beyond 0.5 per cent offset a strain rate of 0.05 ± 0.01 per minute shall be used to fracture.

Note 3. (a) It is generally recognized that maintaining the same strain rate throughout the test is preferable to the above recommended method. However in order to avoid time consuming tests, an increase of the strain rate above 0.5 per cent strain is admitted.

(b) Strain measurement from cross-head movement or strain rate measured from rate of head separation may considerably deviate from actual strain or strain rate. Attention is drawn to the effect of these variations on test results.

1.7 Strain Measurement and Strain Rate above 850°C

1.7.1 Method

The same considerations apply here as in section 1.6.1.

Note 4. In view of the fact that extensometers for service in vacuum of inert gas chambers at temperatures above 850°C are not widely available, strain recording may be made by controlling the cross head speed. This is carried out, preferably, by means of a pacer to provide a controlled rate of head separation in inches per inch of gauge length per minute. Where strain rate is approximated by controlling cross head speed, speed shall be measured under actual loading conditions. Attention is drawn to the remarks of Note 3b.

1.7.2 Strain Rate

The strain rate recommended in section 1.6.2 shall be used.

1.8 Room Temperature Control

All tensile testing shall be conducted in a room whose ambient temperature shall be held within 18 and 30°C. Actual temperature shall be reported. During all tests the range of room temperature should not exceed 5°C. During all tests, the equipment should be shielded from drafts.

1.9 Heating for Elevated Temperature Tests

1.9.1 Temperature Control

The indicated temperature at any point within the gauge length of the test piece shall not vary by more than the following from the nominal test temperature:

Nominal Test Temperature	*Permissible Variation*
Up to and including 1000°C	± 3°C
Above 1000°C up to and including 1500°C	± 10°C
Above 1500°C up to and including 1900°C	± 15°C

At any time during the test the temperature gradient along the gauge length should not exceed 0.5 per cent of the nominal test temperature. Where possible the actual temperature at the point of fracture shall be recorded.

1.9.2 Temperature Measurement

The method of temperature measurement must be sufficiently sensitive and reliable to insure that the temperature of the specimen is within the limits specified in section 1.9.1.

Base-metal thermocouples may be used for test temperatures up to including 1150°C, whereas noble metal couples may be used for test temperatures up to 1500°C.

For test temperatures in the 1350 to 2200°C range, high temperature thermocouples or optical pyrometers may be used.

In all cases data shall be provided to demonstrate the accuracy of the temperature sensor device.

Regarding calibration and attachment of thermocouples, the following requirements shall be followed:

(a) Thermocouples are generally used in conjunction with potentiometers or millivolt meters. Such temperature measurements are subject to two types of errors. Thermocouple calibration and instrument measuring errors initially introduce uncertainty as to the exact temperature. Secondly, both thermocouples and measuring instruments may be subject to variation with time. Consequently the temperature measuring equipment must be periodically checked. Common error encountered in the use of thermocouples to measure temperatures include: calibration error, drift in calibration due to contamination or deterioration with use, leadwire error, error arising from method of attachment to the test piece, direct radiation of heat to the bead, heat-conduction along thermocouple wires, etc..

(b) Temperature measurements shall be made with thermocouples of known calibration. Representative thermocouples shall be calibrated from each lot of wires used for making base-metal thermocouples. Except for relatively low temperatures of exposure, base-metal thermocouples are subject to error upon re-use, unless the depth of immersion and temperature gradients of the initial exposure are reproduced. Base-metal thermocouples shall not be re-used without clipping back to remove wire exposed to the hot zone and rewelding. Any re-use of base metal thermocouples after relatively low temperature use without this precaution should be accompanied by recalibration data demonstrating that calibration was not unduly affected by the conditions of exposure.

Noble metal thermocouples are also subject to errors due to contamination, etc., and should be annealed periodically and checked for calibration. Care shall be exercised to keep the thermocouples clean prior to exposure and during use at elevated temperatures.

Measurement of the drift in calibration of thermocouples during use is difficult. When drift is a problem during tests, a method should be devised to check the readings of the thermocouples on the test piece during the test. For reliable calibration of thermocouples after use, the temperature gradient of the testing furnace must be reproduced during the re-calibration.

(c) In attaching thermocouples to test pieces it is important that the junction be kept in intimate contact with the test piece and shielded from radiation. The bead should be as small as possible and there should be no shorting of the circuit (such as could occur from twisted wires behind the bead). Ceramic insulators should usually be used on the thermocouples in the hot zone. If some other electrical insulation material is used in the hot zone, it should be carefully checked to determine whether the electrical insulating properties are maintained with higher temperatures.

(d) Temperature measuring, controlling and recording instruments shall be calibrated periodically against a secondary standard, such as a precision potentiometer. Leadwire error shall be checked with the lead wires in place as they normally are used.

1.9.3 Heating Rate

The specimen shall be brought to test temperature in not less than five minutes or not more than 60 minutes and held at test temperature for 15 minutes before loading in order to fulfill the temperature requirements as specified in section 1.9.1.

Note 5. The heating rate adopted shall be such as to prevent deviation from particularly the required purity of the test environment (speed of pumping in the case of a vacuum atmosphere). Furthermore, attention is drawn to the possible effect of heating rate on the microstructure of the material undergoing testing.

1.10 Test Environment

All elevated temperature testing, shall be conducted under conditions to ensure that the surface of the specimen will not be chemically affected during the test to an extent which significantly affects strength properties. Vacuum of better than 10^{-4} mm. of mercury is recommended but inert gas atmospheres of equivalent purity may also be used. Eventually, use of protective coatings may be made, provided that adequate supporting data are presented for their effectiveness and their non influencing the mechanical properties measured.

1.11 Post Test-Inspection

Following test, a representative test-piece or test-pieces shall be subjected to the same tests as specified in section 1.4. Changes in chemical analysis, hardness or microstructure shall be reported.

1.12 Reports

The report shall include the following:

(1) description of the material tested;

(2) results of pretest and post-test inspection on representative specimens

(3) description of the test pieces used and method employed for machining them

(4) description of the test equipment:
- type of tensile testing machine and characteristics
- gripping method
- method and accuracy for measuring load, strains and strain-rate
- method of heating and for measuring temperature
- axiality of the machine.

(5) rate of application of load or strain rate below and above 0.5 per cent offset

(6) heating rate and actual time at temperature before loading

(7) stress-strain curve for all individual tests

(8) modulus of elasticity and 0.2 per cent offset yield strength for each test

(9) percentage elongation with gauge length defined, for each test

(10) tensile strength for each test

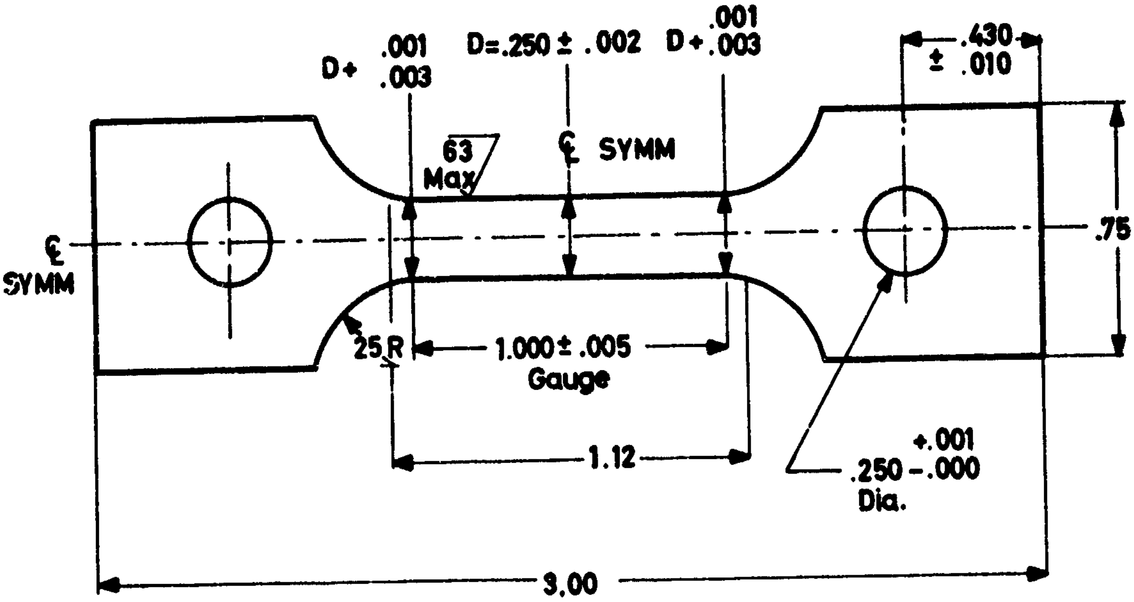
(11) test temperature and temperature variation along the gauge length and during the test.

Note 6. Regarding elongation the following recommendations shall be followed:

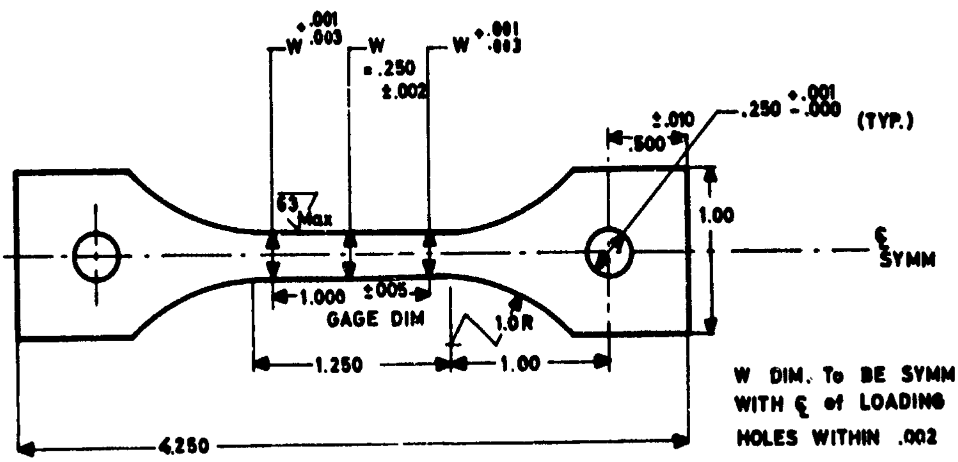
(a) gauge marks punched or scribed on the surface of the test piece prior to testing shall be used to define the gauge length. Punched or scribed gauge marks are not required, however, when such marks will affect the properties of the material being tension tested. When gauge marks on the gauge length cannot be used, marks on the shoulders of the specimen, over-all specimen length or other similar methods can be used to establish the over-all extension of the specimen. The over-all extension can then be used to calculate a percentage elongation after fracture in terms of the initial length of the reduced section of the gauge section. The method used shall be reported with the results.

(b) for measuring elongation the ends of the fractured specimen shall be fitted together carefully and the distance between gauge marks measured to the nearest 0.01 in. elongation shall be reported as the increase in length of the gauge section expressed as a percentage of the original gauge length.

(c) the location and a description of the fracture shall be noted in the report.



(A)



(B)

Fig. 1 Tension test-specimens used for sheet material
(a) typical 1 inch gauge length sheet specimen
(b) typical 1 inch gauge length sheet specimen.

APPENDIX II to APPENDIX A

2. RECRYSTALLIZATION TEMPERATURE DETERMINATION

2.1 Equipment

Conventional diamond pyramid hardness (Vickers or equivalent) and metallographic equipment shall be used to measure extent of recrystallization.

2.2 Test Pieces

Samples of suitable size shall be cut from the "as-received" sheet. Surface conditions shall not be changed in sample preparation.

2.3 Test Procedure

2.3.1 Annealing

All test pieces shall be annealed one hour at temperature in a vacuum or inert atmosphere of sufficient quality to prevent surface contamination. Test pieces shall be heated to annealing temperatures in no less than 5 minutes and no more than 60 minutes. Temperature shall be controlled to within ±5°C of set temperature. Tests shall be made at 25°C intervals to establish recrystallization temperature.

2.3.2 Estimate of Recrystallization

The extent of recrystallization shall be determined by metallographic examination of both transverse and longitudinal cross section at 100 × magnification. The diamond pyramid hardness shall be measured on both surfaces with a minimum of five readings on each side.

2.3.3 Recrystallization Temperature

The recrystallization temperature shall be defined as the minimum temperature for which in one hour both of the following conditions are fulfilled:

(a) the structure is at least 50 per cent recrystallized

(b) the drop in hardness is at least 2/3 of the total drop from as-rolled to fully annealed.

Note 1. The second condition may, in some cases, not be fulfilled although recrystallization occurs. In this case, micrographs and hardness values after several test temperatures shall be given.

2.4 Report Requirements

The following data shall be reported:

- Recrystallization temperature
- Per cent structure recrystallized at 1 hour at recrystallization temperature
- As-rolled hardness
- Fully annealed hardness
- Hardness after 1 hour at recrystallization temperature
- Per cent drop in hardness (per cent of total drop on full annealing) after 1 hour at recrystallization temperature.

Note 2. It is known that contamination of a refractory metal with impurities raises the recrystallization temperature. This property may conveniently serve the purpose of determining the extent of surface contamination.

APPENDIX B

Document used for the Cooperative Programme Or the Vascojet 90 (15CDV6) Steel Sheet

1. INTRODUCTION

The present programme originates from the discussions at the meeting held in Paris the 2nd of July 1965. Delegates of several laboratories participating in the programme were present at that meeting.

The purpose of the programme is to clarify some discrepancies that appeared in the results obtained with the TZM material. Specifically the present programme aims at:

(1) the evaluation of the dispersion of the results occurring during the determination of room temperature tensile-test data under usual conditions.

(2) the evaluation of the dispersion of the results occurring during the determination of room temperature tensile-test data under the conditions used for tests at elevated temperatures.

For the purpose defined in this way, an homogeneous material shall be used. The tensile strength of this material is similar to that of the TZM molybdenum alloy at 1050°C.

2. DEFINITION OF THE MATERIAL

The material considered in this programme was made available to the Materials Group by Sud-Aviation through the courtesy of France. It was carefully selected to insure the homogeneity of the samples offered to the Materials Group.

The material consists of sheets of steel 15CDV6 (Vascojet 90). Each participating laboratory will receive one sheet 500 × 150 × 1.5 mm.

The original sheet was cut by Sud-Aviation according to the scheme reproduced in Figure 1. Furthermore the tensile properties of the material were determined in the laboratories of Sud-Aviation with a Losen tensile machine. The results of these determination are reproduced in Table 1. The position of the test pieces relative to the original sheet is indicated in Figure 1.

3. TESTS

3.1 Test-Pieces

The test-pieces shall be machined out of the sheet according to a predetermined scheme. This scheme aims at obtaining a random distribution of specimens. Figure 2 reproduces the sampling scheme. The piece containing the defect due to the marking shall be replaced by the free-one having the next inferior number. Figure 3 reproduces the scheme for the distribution of the sheets to the different participants.

3.2 Conventional Tensile Tests at Room Temperature

Ten (10) tests shall be carried out according to the recommendations of Appendix I of the document relative to the testing programme on the TZM-alloy sheet. The same techniques and the same procedure shall be used as for the TZM sheet.

3.3 Tensile Tests at Room Temperature under the Conditions Used for Elevated Temperature Tests

Ten (10) tests shall be carried out according to the above mentioned recommendations for the tests at elevated temperature, without, however, heating the specimen. In this case also the same equipment and the same procedure shall be used as for the tests at elevated temperature with the TZM material.

REPORTS

The experimental conditions and all the individual results shall be reported according to the recommendations given in Appendix I of the document mentioned above. A copy of the report shall be forwarded to the coordinator. Thirty (30) copies shall be forwarded to the Executive of the Structures and Materials Panel, for distribution within AGARD.

Liège, February 1966.

D. COUTSOURADIS
Agard Coordinator.

TABLE 1

Data on Original Sheet as Determined by Sud-Aviation

Sheet No.	*0.2 YS kg/mm²*	*U.T.S. kg/mm²*	*Elongation %*
1	48.7 51	63.7 63.2	24 24
2	47.8 50.3	61.2 65.3	22 20.5
4	46 49.2	59.5 63	24 24
5	50.2 51.1	65.3 66.4	22 20.5
6	48.8 48.8	63.3 63.2	22 22
8	50 50.7	65 66	22 22
9	49.3 49.8	64.7 63.4	24 22
10	51.2 50.8	66.8 66.4	20.5 22
11	52.7 49.6	68.4 65.4	26 24
12	48.9 50.2	63 65	24 24
Average	49.25	64.3	22.8

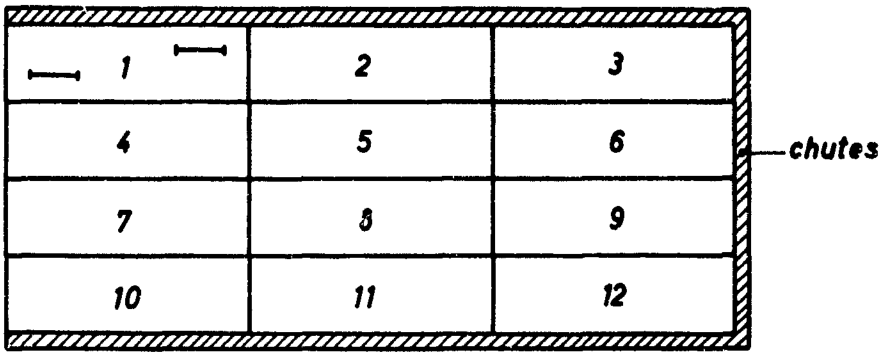
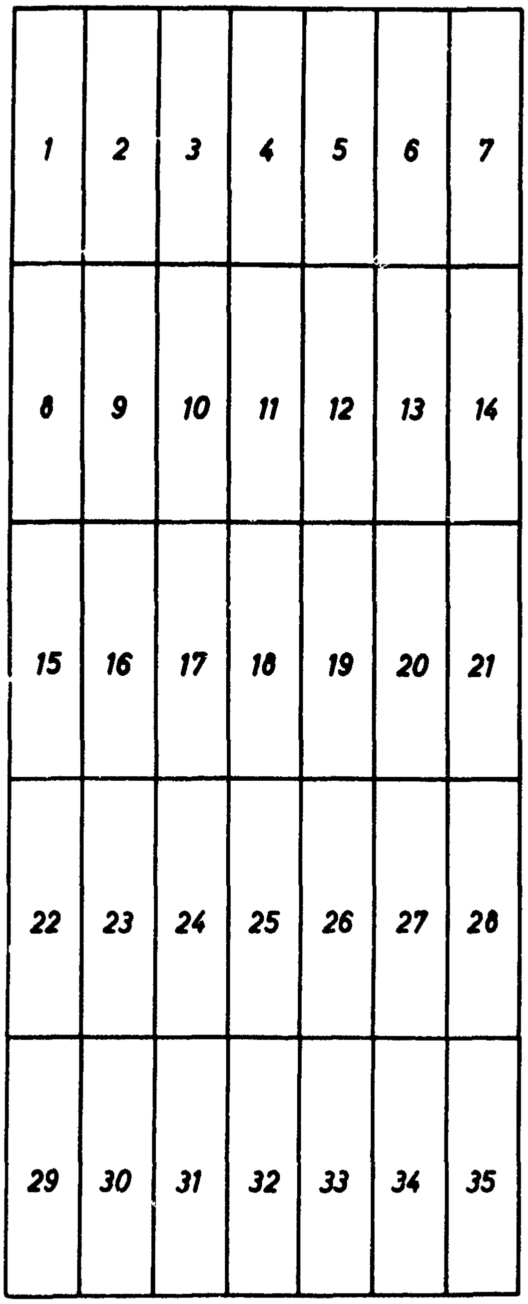


Figure 1



Prélèvement d'éprouvettes sur tôle de 150 x 500 x 1,5 mm

Echantillons:
9-12-16-18-19-26-29-30-31-34
1ier groupe d'essais

Echantillons:
1-2-3-13-15-20-21-27-32-33
2e groupe d'essais

Sampling of test-pieces on Sheet 150 x 500 x 1,5 mm

Test-pieces :
9-12-16-18-19-26-29-30-31-34
1st group of Tests

Test-pieces :
1-2-3-13-15-20-21-27-32-33
2nd group of Tests

Figure 2

APPENDIX C

Evaluation of the TZM Sheet Material by the Producer (Universal Cyclops Steel Coporation)

Refractory Metal Sheet Evaluation

Material: TZM *Producer:* Universal Cyclops Steel Corporation

Heat: KDTZM 1201 *Lot*: 11 *Sheet*: 58 & 58 *Gauge*: 0.60 in.

Manufacturing Data:
Melt 8 in. dia ingot, extrude to 4-1/4 in. dia 2100°F
Anneal 1 hr at 2800°F, in Fab forge at 3400°F to 1.5 in. thick
Anneal 1 hr at 2800°F roll at 2200°F to 760 in. thick
Anneal 1 hr at 2700°F roll at 2200°F to 264 in. thick
Anneal 1 hr at 2150°F cross roll at 1800°F to 0.66 in. thick
Anneal 1 hr at 2300°F belt grind and pickle to 0.60 in. thick.

Specimen preparation:
Room temp. tensile: Mill.250" x 1.0" gauge length with 1 in. radius
2000°F tensile; Grind. 250" x 1.0" gauge length with 1/4 in. radius
Notched R.T. Tensile: Grind. 50" x 1.0" gauge length with 1/4 in. radius and grind notch.
Bend: Grind 1/16 in. min., from edges of blank strip. Abrasive cut length mults. De-burr edges and corners with 120 grit abrasive.

Testing Machine:
R.T. Tensile and all bends: Baldwin-Lima-Hamilton universal testing machine 2000°F tensile: Brew Furnace.

Strain Rate:
R.T. Tensile: 0.05"/"/' to 6% Y.S., .05"/"/' to failure
(Extensometer)
Notched tensile: 0.05"/minute head speed
2000°F tensile: 05"/"/' (Deflectometer)
Bends: 1"/minute ram travel

Test Environment:
2000°F Tensile: Vacuum (5 micron) induction heat to 2000°F in 15 min. and hold 15 min.
Bends: Liquid nitrogen cold chamber, hold at temp. 5 minutes.

Refractory Metal Sheet Evaluation

Heat: KDTZM1201 *Lot*: 11 *Sheet*: 57 *Gauge*: 060

Chemistry:	C	Ti	Zr	Si	Fe	Ni	O_2	N_2	H_2
%	.032	.51	.11	<.0035	<.0015	<.001	.0008	.0003	.0001

Recrystallization:	*End A*	*End B*
As received D.P.H	307	302
Fully Annealed D.P.H.	199	201
Rex.Temp (°F)	2450°	2550°
% Recrystallized:	50%	65%
% Hardness Drop	83%	71%

Room Temp. Tensile	*U.T.S. (KSI)*	*2% Y.S. (KSI)*	*% Elong.*
End A Transverse	132.2	127.2	12.3
End A Longitudinal	129.8	114.6	17.8
End B Transverse	130.3	124.1	12.9
End B Longitudinal	128.5	115.4	15.2

Notched Tensile:	*End A*	*End B*
Longit. Notch Strength:	136.9 K.S.I.	128.6 K.S.I.

2000°F Tensile:	*U.T.S.* (KSI)	*Y.S.* (KSI)	% *Elong.*
End A Transverse	85.0	79.3	7.0
End B Transverse	84.0	80.1	6.8

Bend Tests	*End A(Transv)*	*End B (Transv)*	*End B (Longit)*
4T Minimum Bend Temp	-50°F	-50°F	-75°F
Next Failed Temp.	-75°F	-75°F	-100°F
Min. Bend radius at R.T.	0T	0T	0T
Springback angle	< 5°	< 5°	< 5°
Specimen size	¾" × 3¼"	Fixture Span Length	1½"

Refractory Metal Sheet Evaluation

Heat: KDTZM1201 *Lot*: 11 *Sheet*: 58 *Gauge*: 060

Chemistry:	C	Ti	Zr	Si	Fe	Ni	O_2	N_2	H_2
%	.031	.55	.083	<.0035	<.0015	<.001	.0013	.0003	.00033

Recrystallization	*End A*	*End B*
As received D.P.H.	301	301
Fully Annealed D.P.H.	199	197
Rex. Temp (°F)	2500	2450
% Recrystallized:	50%	50%
% Hardness Drop	68%	67%

Room Temp. Tensile	*U.T.S.* (KSI)	2% *Y.S.* (KSI)	% *Elong.*
End A Transverse	134.9	127.4	13.0
End A Longitudinal	128.4	112.2	14.5
End B Transverse	134.3	126.4	13.1
End B Longitudinal	122.5	112.0	18.7

Notched Tensile:	*End A*	*End B*
Longit. Notch Strength	133.5 K.S.I.	127.5 K.S.I.

2000°F Tensile:	*U.T.S.* (KSI)	*Y.S.* (KSI)	% *Elong.*
End A Transverse	82.0	77.8	6.4
End B Transverse	79.4	75.1	6.6

Bend Tests	*End A (Transv)*	*End B (Transv)*	*End B (Longit.)*
4T Minimum Bend Temp.	-100°F	-100°F	-125°F
Next Failed Temp	-125°F	-125°F	-150°F
Min. bend radius at R.T.	0T	0T	0T
Springback angle	< 5°	< 5°	< 5°
Specimen size	¾" × 3¼"	Fixture Span length	1½"

Evaluation of the Homogeneity of TZM sheet

The evaluation of Homogeneity of TZM sheet was made on the basis of DOTSON'S data*. These refer to 0.060 in. thick sheets originating from two different heats. The longitudinal 0.2% YS at RT and at 2200°F (1200°C) from specimens from the center and corner of the sheets is reproduced in Tables 1 and 3.

* Reference (1) in main report.

TABLE 1

RT Longitudinal 0.2% YS of 0.060 in. thick TZM alloy sheet (in kg/mm^2)

	Center	Edge	
Heat G	78.8 79.2 79.9	79.4 78.0 79.1	
	237.9	236.5	474.4
Heat H	79.2 78.5 78.6	78.3 78.2 78.3	
	236.3	234.8	471.1
	474.2	471.3	945.5

TABLE 2

Analysis of Variance of Table 1

Source	S.S	d.f.	MS	F			
						(95)	3.46
Between Position	BCSS 0.70	1	0.70	2.8	F(1.8)	(99)	5.32
Between Heats	BRSS 0.91	1	0.91	3.64		(99,9)	11.30
Interactions	ISS 0.00	1	0.00				
Within Cell	WSS 2.00	8	0.250				
Total	TSS 3.61	11					

TABLE 3

2200°F Longitudinal 0.2% YS of 0.060 in. TZM Alloy Sheet (kg/mm^2)

	Center	Edge	
Heat G	49.3 48.6 49.4	48.9 48.4 49.2	
	147.3	146.5	293.8
Heat H	44.9 38.2 47.1	46.5 46.5 46.6	
	130.2	139.6	269.8
	277.5	286.1	563.6

TABLE 4

Analysis of Variance

Source	S.S.		d.f.	MS	F
Between Position	BCSS	6.164	1	6.164	2.2
Between Heats	BRSS	48.000	1	48.000	14
Interactions	ISS	8.670	1	8.670	3
Within Cell	WSS	43.693	8	2.731	
Total	TSS	106.527	11		

Examination of Tables 2 and 4 relating to the analysis of variance shows that the variation from center to edge of 0.2% YS at RT as well as at 2200°F is not significant. The between heats variability of RT 0.2% YS is slightly significant at the 5% level. That of 2200°F 0.2% YS appears highly significant (0.1% level).

The between sheets variability was evaluated on the basis of data reproduction Table 5.

TABLE 5

RT 0.2% YS (Edge)

	Sheet 2	Sheet 3	
Heat G	79.4 78.0 79.1	80.9 77.2 79.4	
	236.5	237.5	474.0
Heat H	78.3 78.2 78.3	75.0 78.3 75.4	
	234.8	228.7	463.5
	471.3	466.2	937.5

TABLE 6

2200°F 0.2% YS (Edge)

	Sheet 2	Sheet 3	
Heat G	48.9 48.4 49.2	45.1 45.4 43.4	
	146.5	133.9	280.4
Heat H	46.5 46.5 46.6	42.0 41.5 43.7	
	139.6	127.2	266.8
	286.1	261.1	547.2

TABLE 7

Analysis of Variance Table 5 (RT 0.2% YS)

Source	S.S.	d.f.	MS	F
Between Sheets	BCSS 2.16	1	2.16	-
Between Heats	BRSS 9.18	1	9.18	3.98
Interaction	ISS 4.21	1	4.21	1.82
Within Cells	WSS 18.51	8	2.31	
Total	34.06	11		

TABLE 8

Analysis of Variance Table 6 (2200°F 0.2% YS)

Source	S.S.	d.f.	MS	F
Between Sheets	BCSS 47.52	1	47.52	71.0
Between Heats	BRSS 10.85	1	10.85	16.2
Interaction	ISS 4.57	1	4.57	6.8
Within Cells	WSS 5.34	8	0.67	
Total	TSS 68.28	11		

The between sheets variability of RT 0.2% YS is not significant (Table VII) whereas that of 2200°F YS is highly significant.

APPENDIX D

Summary of Modified Specifications

1.1 Test-Piece Dimensions (NM).

	Inches	mm
Gauge length	1.000 ± 0.003	25.40 ± 0.08
Width	.0.250 ± 0.005	0.35 ± 0.127
Radius of Fillet	0.250 min.	6.35 min.

1.2 Gripping Method (NM)

Provided that no bending stresses are introduced, any gripping device is acceptable. Pin loading has proved satisfactory in many cases.

1.3 Surface Finish

The gauge section surface finish shall be the as delivered mill finish. The edges of the gauge section shall be finally polished with 00 emery with the scratches parallel to the low direction. Material damaged by previous machining operations, including eloxing, along the gauge length or around the pin holes shall be removed by milling or other adequate means.

1.4 Pretest Inspection (NM)

The examination will include microhardness measurement, metallographic examination, chemical analysis for interstitial elements and inspection for the presence of cracks.

1.5 Loading Apparatus and Methods (NM)

The type of the machine is not specified. Evidence shall be given for the accuracy and precision of the testing equipment.

1.6 and 1.7 Strain Measurement and Strain Rate

1.6.1.- 1.7.1 Method

An extensometer shall be used to determine strain from zero to about 0.5 per cent offset. For Young's modulus determination the maximum error of the extensometer shall be of 0.01 per cent or less. For total elongation measurements gauge marks may be used unless they affect the material fracture behaviour.

1.6.2 - 1.7.2 Strain Rate

A strain rate of 0.005 ± 0.001 per minute shall be used to 0.5 per cent offset. Beyond 0.5 per cent offset a strain rate of 0.05 ± 0.01 per minute shall be used to fracture.

Note. When the determination of Young's modulus is not requested, strain may be approximated by cross head travel. Strain rate may be approximated by cross head speed. It must however be recognized that the variability may slightly increase or that a small systematic error may affect the results.

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* NM represents non modified recommendations relatively to the original specification given in Appendix A.

1.8 Room Temperature Control (NM)

The control temperature shall be held within 18 and 30°C. During tests the maximum variation of room temperature should not exceed 5°C.

1.9 Heating for Elevated Temperature Tests

1.9.1 Temperature Control

Nominal Test Temperature	*Variation*
Up to and including 1000°C	± 3°C
Above 1000°C up to and including 1500°C	± 8°C
Above 1500°C up to and including 1900°C	± 12°C

During the test the temperature gradient along the gauge length should not exceed 0.5 percent of the nominal test temperature.

1.9.2 Temperature Measurement (NM)

Up to 1150°C Chromel-Alumel thermocouples are suitable. Up to 1500°C Pt-PtRh thermocouples may be used. For higher temperatures W-WRe, Mo-W, W-Ir thermocouples or optical pyrometers are useful. The latter should be carefully calibrated for emissivity changes, absorption in the windows etc. Among different thermocouples preference should be given to those giving the largest emf output to temperature ratio. Other factors of selection include cost, convenience of assembling, reaction with insulators, etc.

1.9.3 Heating Rate

The specimen shall be brought slowly to 450°C in order to maintain a vacuum of better than 10^{-4}. From 450°C to the test temperature the heating rate shall be as closely as possible of 20°C per min. The specimens will be held at the test temperature for 15 minutes before loading.

1.10 Test Environment

Vacuum of better than 10^{-4} mm Hg is recommended for uncoated refractory metals. In particular cases a better vacuum should be achieved as necessary.

1.11 Post-Test Inspection (NM)

Following test the same examination should be carried out on a representative test-piece as recommended in section 1.4.

1.12 Reports (NM)

All data relevant to the equipment, test conditions and test results shall be reported.

APPENDIX E

Experimental Techniques

In this appendix the experimental techniques used by the participating laboratories are described in extenso as given in the individual reports.

1. LABORATORY I

1.1 Recrystallization Temperature

Specimens with dimensions of 10 × 20 mm are annealed in a horizontal "crusilite" resistance furnace under a vacuum of 10^{-4} mm Hg. The time for reaching the regime temperature varies between 40 and 60 minutes for temperatures ranging from 1000 to 1500°C. As specified a constant temperature (±5°C) is maintained for 1 hour.

After thermal treatment, the recrystallization is followed by microscopic examination at a magnification of 100 times, and by microhardness measurements by means of an "Otto Wolpert" apparatus using a load of 20 kg.

The recrystallization temperature is taken to be the temperature at which the above heat treatment causes:

(1) partial recrystallization of minimum 50%, as estimated microscopically,

(2) decrease in Vickers hardness of at least 2/3 of the total hardness drop caused by complete recrystallization.

1.2 Tensile Tests

Tensile tests on the TZM alloy were performed at room temperature and at 1050 and 1450°C by means of a 5t "Instron" testing machine, type TT-Floor Model. For the high temperatures the machine was equipped with a "crusilite" resistance furnace with vacuum chamber.

Test pieces were milled in the longitudinal direction of the sheet. Before the test all specimens were checked for cracks by fluorescent penetrant inspection; defective specimens were eliminated.

During the tests a constant cross-head speed of 0.1 mm/min., corresponding to a strain rate of 0.004/min, was maintained, except for a few tests at higher temperatures where the strain rate was increased by a factor ten after a strain of 0.5 per cent had been reached.

The test temperature was measured by means of a Pt/Pt 10Rh thermocouple, for the last high temperature tests by a Pt5Rh/Pt20Rh thermocouple. The temperature was kept constant within ±3°C of the nominal test temperature. Reaching the regime temperature needed about 1.5 hour for 1050°C, and 2 hours for 1450°C; before loading, the specimen was held at test temperature for 15 minutes. During heating a dynamic vacuum between 10^{-3} and 10^{-4} mm Hg could be held; testing was performed under a vacuum of 10^{-4} to 2×10^{-5} mm Hg.

Conventional room temperature test for Vascojet 90 steel sheet were carried out with the same machine as above, using a wedge-grip system. The tests under high temperature conditions were carried out with a pin and hole grip system.

2. LABORATORY II

2.1 Recrystallization Temperature

A first series of specimen were sealed under argon in quartz tubes, heat treated for one hour at 1200, 1250, 1300 and 1350°C, then quenched. Other specimens were treated for one hour at 1375 and 1400°C in an induction furnace.

After treatment the specimens were electrolytically polished in a mixture of H_2SO_4 + HCl + CH_3OH and then etched in a solution containing:

1 vol.HF, 1 vol.H NO_3, 2 vol.HCl .

The specimens were then examined with the light microscope. The Vickers hardness was measured with a load of 10 kg by means of a Wolpert durometer.

2.2 Tensile Tests

2.2.1 Room Temperature Tests

A series of tests was carried out on specimens from sheet No. 18 with a hydraulic 50 T Baldwin machine. The results of these tests are reported under column II(b) in Tables 17 to 19. With this machine the accuracy of load measured was of ±1%. A low magnification (×20) extensometer was used, and thus the strain rate could not be controlled adequately. The loading rate was instead controlled at the value of 400 kg/min. All other tests were carried out with an Instron tensile machine, type TT-DM-L, of 10,000 kg capacity. Load measurement was made by means of strain gauge load cells. One of those cells was of 10,000 kg capacity and had the following working ranges: 0-200, 0-500, 0-1000, 0-2000, 0-5000 and 0-10,000 kg. A second cell covered the ranges: 0-10, 0-20, 0-50, 0-100, 0-200 and 0-500 kg. In both cases the accuracy of load measurement was of ±0.5% of the indicated value or ±0.25% of the scale whichever was the larger.

The load cells were calibrated by means of dead weights. Furthermore, with the 10,000 kg load cell it was possible to calibrate electronically the load cell before each test. For all tests in vacuum, the zero setting of the system, was made after the required vacuum was reached.

Strain was measured by means of Instron strain-gauge extensometers with a gauge length of 25 mm and an elongation capacity of 10%. This extensometer permitted a magnification of 1,000 times over the first 1% strain, 500 times over 2%, 200 times over 5% and 100 times over 10%.

The system consisting of the extensometer and XY recorder was calibrated by means of a micrometric device with a sensitivity of 1 micron. Under these conditions, the strain measurement for the determination of yield strength was carried out with an accuracy of ±0.004% which corresponds to a 1 mm length on the paper when using the 25 mm extensometer with a magnification of 1,000 times. The strain rate was controlled by means of the Instron "Strain pacer", for strain up to 0.5% at a value of 0.004/min which was within the specified range. Beyond 0.5% strain the extensometer was removed; strain rate was then controlled by means of a constant cross-head speed of 2 mm/min. The latter was chosen considering that the fictitious gauge length of the specimen was of 33 mm.

2.2.2 Elevated Temperature Tests

These were carried out by adapting on the Instron machine a Brew furnace with Tantalum resistors capable of yielding temperatures up to 2500°C. During the tests the vacuum was improved from, generally, 7×10^{-5} Torr at the beginning to 10^{-5} Torr at the end of the test. Since no extensometer was available the elevated temperature tests were carried out with constant cross head speed of 0.2 mm/min. till 0.5% strain and 2 mm/min. from 0.5% strain

to fracture. These speeds were chosen by taking into account the limitations of the machine, the specified strain rate range and the same fictitious gauge length of 33 mm as for the room temperature tests.

At 1040 and 1450°C the pulling rods and pins were made of TZM alloy, whereas at 1800°C these pieces were of thoriated tungsten.

Heating time varied from 45 min. to 1 hour depending on a more or less large degassing rate up to 500°C. During heating to the desired temperature a load of approximately 4 kg was maintained on the specimen. An automatic device maintained this load constant and therefore, compensated for the thermal elongation of the specimen. After reaching the specified temperature, the latter was stabilized for 15 min.

Temperature for tests at 1050° and 1450°C was measured with Pt-Pt10Rh thermocouples attached to the specimen by means of quartz rope. At 1800°C W-Mo thermocouples were used. The 0.5 mm wire lot of these thermocouples was calibrated against a W-Ir thermocouple which was calibrated by the producer. Welding was carried out with an electric arc between tungsten electrodes under argon. The calibration tests showed a good reproducibility of the EMF output for different couples and also a good stability during at least 3 heating cycles.

The temperature gradient along the gauge length after stabilization for 15 min. was of less than 4°C at 1050°C and less than 3°C at 1450°C. At 1800°C the measurement of the gradient was not made but it is estimated that it was at least as good as at 1450°C.

For all tests, including those carried out at room temperature, 0.2% yield strength and ultimate strength were measured on the stress-strain curve; elongation was measured by means of an optical comparator on markings traced at a distance of 25 mm.

The test pieces of TZM alloy were first cut with an electrospark machine and then finish machined by milling. Longitudinal edges were furthermore polished with 00 Emery paper. Vascojet 90 steel specimen were prepared in the same way except that the initial cut was carried with the saw.

3. LABORATORY III

3.1 Tensile Tests on TZM Sheet

3.1.1 Loading Apparatus

The apparatus used for the application of load is a 10,000 lb capacity Instron testing machine. This machine incorporates a highly sensitive electronic weighing system; employing bonded wire strain gauges, and a Leeds and Northrup X-Y recorder for detecting and recording the tensile load applied to the sample under test.

The error in the load detecting system is stated by the manufacturer to be less than ±0.5% of the load range.

The calibration of this system was checked periodically with dead weights.

The chart of the recorder is driven synchronously at various speed ratios with respect to the cross-head, so that load-elongation diagrams may be obtained with a large choice of magnification factors without the use of an extensometer.

The gripping adaptors were slotted for pin connections. These adaptors were machined from molybdenum - 0.5% titanium material, and were used up to temperatures of 1800°C (3272°F). The pins were machined from tungsten. Some difficulty was encountered in earlier tests where the pins were made from the same material as the adaptors (Mo-0.5Ti). At the

higher temperatures, pins of this material deformed and pressure welding occurred between the pins and the adaptors. This made the removal of the broken specimens difficult. However, as mentioned, this situation was remedied to a great extent with the use of tungsten pins.

To ensure that the test specimens are loaded axially, the center line of the vacuum furnace is carefully aligned with the loading adaptors located in the cross-heads of the testing machine. This is accomplished by passing a straight steel bar through the specimen pull rod ports in the furnace and adjusting the support brackets, used to attach the furnace to the testing machine, until the steel bar could be fitted freely into the loading adaptors.

3.1.2 Vacuum Assembly

The Brew Model 1060 high vacuum furnace was designed for use with loading apparatus such as the Instron machine and may be used up to temperatures of 2500°C (4532°F). The heating element, 2 inches in diameter and 6 inches high, was fabricated from tantalum sheet material. The heat shields were also made from tantalum sheet of 0.005 inch thickness.

The upper and lower pull rods, which passed through the sealed ports of the furnace, were made of stainless steel and were cooled by circulating water. These pull rods were attached to the upper and lower loading adaptors.

The vacuum pumping system consisted of a 4-inch oil diffusion pump manufactured by Consolidated Vacuum Corporation, USA. This pump was backed by a compound mechanical pump with a rated ultimate pressure of 0.2 microns and an air displacement of 5-8 cubic feet per minute. The system also contained a nitrogen cold trap located in the vacuum manifold between the high vacuum valve and diffusion pump.

The pressures in the foreline and vacuum chamber were measured by thermocouple gauges with a range of 1000 microns to 1 micron. For lower pressures a Veeco Vacuum ionization gauge was used to measure the pressure in the vacuum chamber.

3.1.3 Temperature Measurement and Heating

The specimen temperature was measured by a Leeds and Northrup disappearing filament optical pyrometer. It was assumed that black body conditions were approached during these temperature measurements; the sample being sighted through a small opening in the heater shell and heat shields. Therefore, the brightness temperature was considered to be the true temperature.

The optical pyrometer was checked for brightness temperature condition by means of a special Calibration Bulb manufactured by the General Electric Cy.

The heating of the specimen was carried out progressively for ¾ hour to 1 hour depending on the outgassing on the heating elements, shields and specimen. As the outgassing depended on the heating rate, the power input to the furnace was controlled at a level such that a vacuum of 10^{-4} mm Hg or better would be maintained.

3.1.4 Machining and Pretest Inspection

Some difficulty was encountered in cutting the sample blanks. The initial attempt to use the shearing for cutting was discontinued because the material was too brittle and seemed to crumble under the knife edge. The blanks were cut by means of a band saw. This method was successful. The specimens were then machined to the final dimensions by means of a milling cutter. The edges were then carefully deburred and polished longitudinally with 00 emery. The gauge section surface finish was as-delivered mill finish.

All specimens before testing were carefully checked by a liquid dye penetrant (ARDROX) for surface cracks. It was found that some specimens after machining contained surface cracks that progressed across the width of the gauge length, whereas others were cracked near the gripping ends. These specimens, in most cases, were cracked only partially through the thickness. However, they were rejected for test.

Five hardness readings were taken at each end of the specimens using a Vickers hardness tester with an applied load of 10 kg.

3.1.5 Test Procedure

The specimen and adaptors were cleaned with acetone prior to and after the specimen was placed in the test chamber. The furnace tank was then connected to the mechanical pump for roughing. When the pressure fell to less than seventy five (75) microns, the diffusion pump was placed into operation. After the test chamber was evacuated to about 10^{-4} mm Hg pressure, the specimen was brought slowly to test temperature. The heating was manually controlled at a rate such that outgassing of the heater assembly and specimen did not cause the pressure to rise above 10^{-4} mm Hg. The time required to reach the test temperature varied from ¾ hour to 1 hour depending on the temperature level. The specimen was then held at test temperature for 15 minutes. The cross-head speed during the initial loading up to 0.5% offset was 0.005 in/min. after which the speed was increased to 0.05 in/min. to fracture. The cross-head movement was changed without interruption of the test by moving a lever which provided the required 10:1 speed ratio. The pull-in force exerted by the vacuum on the top specimen adaptor was balanced out prior to loading the specimen. Hence, the load indicated by the X-Y recorder was the actual load applied to the specimen during the test.

The bottom specimen adaptor was provided with an expansion fixture within the vacuum chamber. This fixture prevented the specimen from being subjected to compressive loads resulting from pull-in of the adaptors during vacuum pump down and expansion of the specimen during heating.

An extensometer for strain measurement at elevated temperatures was not available for use with the furnace. Therefore strain measurements were determined from the cross-head movement of the machine. Since the gauge length of the sample was 1 inch the strain rates based on cross-head speeds were 0.005 in./in./min. and 0.05 in./in./min.

In view of the deformation of the specimen surfaces as evidenced in previous tests on refractory metals, and because of the brittleness of the TZM material at room temperature, it was considered that gauge marks punched or scribed on the surface of the specimens would either be difficult to detect or become a source of premature fracture. Therefore, the elongation was determined by measuring the minimum distance between the pinholes in the grip ends of the specimens before and after fracture. The overall tension was used to calculate the per cent elongation after fracture in terms of the initial length of the gauge section.

3.2 Tensile Tests on Vascojet 90 Sheet

3.2.1 Conventional Tensile Tests at Room Temperature Outside the Vacuum Chamber

For these tensile tests, the furnace and vacuum diffusion pump were swung away from the Instron testing machine. The specimens were secured with pins in the slotted loading adaptors. The top adaptor was attached to the load cell through a universal joint, while the bottom adaptor was provided with a ball and socket connector to ensure axial alignment.

The temperature of the room during all test was approximately 78°F.

A cross-head speed of 0.005 inches per minute was maintained up to an offset of 0.5%. Thereafter, the speed was increased to 0.05 inches per minute until fracture occurred.

In all tests, the percentage elongation was determined over a one inch gauge length, lightly scribed on each specimen.

3.2.2 Tensile Tests at Room Temperature Under Elevated Temperature Conditions

The same procedure was followed for this group of tests as had been carried out in part 3.2.1 with the exception that each specimen was placed in the test chamber under a vacuum of approximately 4×10^{-5} mm Hg.

4. LABORATORY IV

4.1 Specimen Configuration

The elevated temperature specimens were 10 in. long and 0.5 in. wide. The room temperature specimens had:

Total length: 8 in.

Gauge length: 2.5 in.

Width at gauge length: 0.5 in.

Width at grips: 0.750 in.

Grip length: 2.5 in.

Length at fillet: 0.25 in.

The sizes of the specimens are much larger than those recommended for tests using conventional furnace type heating. The advantages to be expected when using the smaller specimens do not apply when the self-resistance type of heating is utilized. In fact, the smaller samples can be a disadvantage due to temperature control. The specimens were machined using conventional techniques.

4.2 Test Equipment

These tests were performed using the company designed and built resistance heated elevated temperature test unit. A detailed discussion of the unit can best be made by breaking the unit into its component parts including the temperature controller, and power regulator... the test unit... and the load time, load strain, strain time and temperature time recorders.

The geometry of the specimen is quite simple, consisting of a 0.500 inch wide and 10 inch long strip of material with sides parallel within ±0.002 inch or a 0.250 inch diameter rod. Unique company developed grips are made such that the serrations bite into the specimen with increasing force with increased load on the specimen. The advantages of such a specimen are, obviously, low material and machining costs. The specimen is horizontal to eliminate the "chimney" heating effect and the length of the coupon assures temperature uniformity across the 2 inch gauge length.

The grips are designed for loads up to 10,000 lb. and are capable of withstanding a 40% overload. Power is fed through leads into the grips which are electrically insulated from the hydraulic loading mechanism and rigid test stand. The extensometer is a lightweight, carbide-tipped, strain-measuring instrument designed by the company. It consists of linear variable differential transformers which sense the strain over the 2-in. gauge length. Counterbalanced extension arms transmit the strain to the differential transformers below the specimen to eliminate the effect of heat from the coupon.

Thermocouple attachment is made by spotwelding appropriate thermocouple combinations directly to the coupon. Connections are available for as many as five separate thermocouples for temperature measurement and control on the specimen. Loading is applied through a servo-controlled hydraulic unit. The load is measured with a ring dynamometer. Various combinations of load dynamometers and hydraulic cylinders are available to increase the accuracy of testing by selecting the unit with the highest response for a particular set of conditions.

A Research inc. temperature controller and power regulator is used to control and programme temperatures. This unit is a complete three-phase ignitron power control unit in combination with three direct current potentiometric circuits and high gain amplifiers. With this unit, heating rates, soaking times, or any time-temperature combination desired can be programmed and automatically controlled.

Strain is sensed by the extensometer and converted into an electrical signal through the linear variable differential transformers. This differentiated position signal is hooked into a feed back system which controls the servo valve of the hydraulic system. With this closed loop feedback system, it is possible to programme the desired strain rate of any particular test from 0.00005 to 0.1 in. per in. per sec.

The load measuring system is also a closed loop servo-controlled feedback system. Loads can be programmed and controlled much the same as strain.

For each test run, a complete load-strain curve is automatically recorded from which the mechanical properties can be obtained. These include ultimate tensile strength, yield strength, and modulus. In addition, records are made of the load-time, and strain-time conditions throughout the test. Due to the over-heating of a resistance-heated specimen when necking down, elongation values generally are not reported.

Tolerances on the test unit are as follows:

load sensing	±.5% of range (i.e. ±.12, ±.5 ± 2.5 ±10 lb. ±50 lb.)
load recording	±.25% of recorder range
strain sensing	±1% or ±100 μin./in. whichever is greater
strain recording	±.25% of recorder range.
temperature control	±½% or ±10°F indicated
Step load application time	.1 sec. minimum, 12 hour maximum
Load range	adjustable 0 to 500% of any range per second
strain rate	adjustable 1 to 5,000 μin./in./sec.
Crosshead speed	0 to 6 in. per sec.
load stability	±.25% of range
load rate repeatability	±5%
strain rate repeatability	±5%
Specimen thickness measurement	±.0002
specimen width measurement	±.0002.

Temperature variation during the greater part of the individual tests was ±5 deg C, but a variation of about 20 deg C was recorded just before fracture in several of the 1450°C tests; this was a consequence of the higher elongation values in the 1450°C series.

Rupture elongation values were obtained from measurements between small, jig-made, marks which defined the gauge length.

7.5 Recrystallization Temperature of TZM

Specimen blanks approximately ½" x ¼" were cut from the material remaining after manufacture of the tensile specimens. After degreasing in acetone, a specimen was placed on either side of the hot junction of a Pt/Pt 13% Rh thermocouple, covered with 0.002 in. thick tantalum foil and bound with molybdenum wire. The tantalum foil acted as a radiation shield and a protective getter for the TZM specimen. The complete assembly was mounted on a molybdenum/stainless steel rod which could move through a gas-tight seal at one end of a platinum wound furnace against a flow of 99.999% argon.

After remaining in a cool (100 to 150°C) part of the furnace for 10 minutes to permit purging of entrapped air, the specimens were passed into the hot zone where the temperature was monitored by a second thermocouple. Within 5 minutes the specimen reached test temperature. During the one hour soaking period, a temperature variation of ±2 deg C was recorded. The assembly was withdrawn to the cooler part of the furnace, and the temperature fell to 100 to 150°C within ten minutes.

Longitudinal and transverse sections of the specimens were prepared. After etching by swabbing with a solution of 10 g $K_3Fe(CN)_6$; 10 g NaOH; 80 ml water, an estimate of the amount of recrystallization was made using a magnification of 100.

8. LABORATORY VIII

8.1 Specimens

Blanks 20 mm wide and 77 mm long were cut with a hand saw made of a tool steel containing 12% Co. Shearing was not retained, because it resulted in severe cracking. The test pieces had the following dimensions:

total length	76.2 mm
gauge	25.4 mm
width at gauge length	6.35 mm
fillet radius	8.00 mm
pinhole diameter	8.00 mm
width at grip head	19.00 mm.

In comparison to the dimensions suggested in the AGARD document, the pin hole diameter was increased. Previous tests had in fact shown that the pin hole diameter should be higher than the gauge length in order to avoid distortion of the hole during the test. Furthermore the fillet radius was increased to 8 mm to insure a more progressive width variation.

The specimens were machined from the blanks by milling, using tungsten carbide tools.

Pin holes were first drilled with a calibrated fixture assuring a perfect reproducibility. The specimens were then machined by 5 on a fixture allowing the obtention of a good axiality of the holes relatively to the gauge length. The surface finish at the gauge length was the delivered surface one: no grinding, machining or etching was carried out. The edges of

5.2.2 Room Temperature Tests-Conventional Equipment

All TZM and steel specimens were tested in the as-milled condition without any special finishing treatment. Tests were carried out using an Amsler tensile testing machine which was known to conform to Grade A requirements of BS 1610:1964. Specimen extension up to the 0.2% proof stress was measured using an optical extensometer of the Lamb roller type having a gauge length of 1 in. and a strain sensitivity of 10^{-5} in./in. Strain pacing equipment was fitted to the testing machine, and ensured that strain rates up to the proof stress fell within the range 0.004-0.005/min. When specimen extension was slightly greater than that needed to define the 0.2% proof stress, the extensometer was removed and the test piece was pulled to fracture. Cross-head speed was increased for this part of the test, but was controlled so that average strain rate values fell within the range 0.06-0.10/min.

5.2.3 Elevated Temperature Tests

Tests at 1050°C and 1450°C were carried out using a modified vacuum stress-rupture machine. It was impractical to incorporate an extensometer in this equipment, and an indication of specimen extension was obtained by reference to cross-head movement, this being measured using the proving ring which had been calibrated by means of dead weights. All tests were carried out using two cross head speeds, the first -0.005 in./min. - being employed until the dial gauge indicated a non-proportional extension of 0.01 - 0.02 in. (this being somewhat greater than the 0.2% proof stress), whilst a speed of 0.05 in./min. was employed from proof stress to fracture.

Attempts were made to comply as closely as possible with the heating rate requirements of MGRT/304; i.e. that specimens should be brought to the test temperature in not more than 60 min. without the pressure inside the vacuum chamber exceeding about 10^{-4} mm Hg. However, because of the characteristics of the vacuum pumps fitted to the equipment; it was impossible to achieve these conditions, and the following technique was adopted. A specimen was set up in the apparatus, the vacuum pumps were switched on, and a small current was passed through the furnace. These conditions were maintained overnight, during which time specimen temperature had risen to about 400-450°C. Furnace input was then gradually increased and it was found that temperature could be raised to 1050°C in about 1¼ h., with a maximum internal pressure of 2×10^{-4} mm Hg. Using this technique, the average time to reach 1450°C was about 1¾ h. with a maximum internal pressure of 2×10^{-4} mm Hg. When the required test temperature had been reached, this was held constant for 15 min. before the specimen was loaded. During this soaking period, internal pressure fell to between 10^{-4} and 4×10^{-5} mm Hg.

Specimen temperature was measured using a potentiometer in conjunction with two platinum-platinum/13% Rhodium thermocouples, these being attached by means of fine tantalum wire to each end of the 1 in. (25.4 mm) gauge length. At 1050°C, the average temperature of the 10 specimens tested fell within the range 1054 ±3°C, the corresponding range for 10 tests at 1450°C being 1446-6°C. During the actual test period, which occupied about 13 min. at 1050°C and 15 min. at 1450°C, these temperature limits were maintained and the corresponding average gauge length temperature gradients were about 15°C and 10°C respectively.

5.2.4 Control Room Temperature Tests on Steel Specimens

Part of the test programme required the testing of steel specimens at room temperature, but using the same apparatus and techniques as those employed when testing the TZM sheet at elevated temperatures. Accordingly, 10 steel specimens conforming to the dimensions given in section 5.2.1 were tested in the modified vacuum stress-rupture machine using the cross-head speeds and measuring equipment described in the previous section of this report.

6. LABORATORY VI

6.1 Tests on TZM Molybdenum Alloy Sheet

6.1.1 Test Specimens

Standard specimens as required by the programme were used. For the room temperature tests, the grip areas were lengthened by 0.5 inch to facilitate use of an extensometer. The gauge section of all specimens was milled to shape and then polished longitudinally with 000 emery to remove milling marks. The surface finish was used as delivered.

Recrystallization test specimens were in the form of 0.75 by 0.75 inch coupons. Each test run used two of these coupons wired together by platinum wire with a thermocouple between them.

6.1.2 Heating Method and Temperature Measurement

For both the recrystallization tests and the elevated temperature tensile tests, a vacuum furnace with a tantalum resistance heating element was used.

The heating rate used was not held constant since it sometimes was necessary to reduce the heating due to outgassing which caused the pressure to rise above 10^{-4} microns. In general, this occurred at temperatures below 1000°C. Above this temperature the heating rate was kept at roughly 60°C per minute.

Temperature measurement was primarily by means of platinum-platinum 10% rhodium thermocouples. However, an optical pyrometer was used to measure variation of temperature along the gauge length of the tensile test specimens at 1050°C and 1450°C and as the only temperature measuring device at 1800°C. Two precision potentiometers were used to measure the E.M.F. of the thermocouples. During the recrystallization tests, the optical pyrometer was calibrated against the thermocouples. The calibration curve was then extrapolated to 1800°C and the value obtained was used for the tensile tests at that temperature.

The temperatures for all tests were kept within the permissible variation about the nominal test temperatures. At 1450° and 1800°C, the temperature gradient along the gauge length was kept within 0.5% of the nominal test temperature, however, at 1050°C the permissible variation was of the same order as the uncertainty in the optical pyrometer measurements and thus, the variation may have slightly exceeded 0.5%.

For all tensile tests, the specimens were held at temperature exactly 15 minutes before loading.

6.1.3 Test Environment

Both the tensile test specimens and the recrystallization samples were heated in vacuum. At all times during heating, the pressure was kept below 1×10^{-4} mm of mercury.

6.1.4 Description and Characteristics of Tensile Test Equipment

The tensile test machine used was an Instron 10,000 pound capacity model with constant cross-head speed. In machines of this type, load is measured by a load cell to which the pull rod is directly attached. Calibration is by means of dead weights which are hung on the pull rods. The machine was calibrated once each day of testing.

Specimens were held by 0.25 inch molybdenum pins thus assuring axiality of loading in one plane. To check axiality in the other plane, dummy specimens were made with strain gauges on each side. Load-extension readings were made, the specimen was rotated 180°

and comparison readings were taken. These tests gave identical results, thus showing axial loading in this plane also. The pull rods were then indexed in order to be sure that the actual specimens were loaded in the same manner as the dummy specimens.

Strain rate was controlled by cross-head motion for all tests. A cross-head speed of 0.005 inches per minute was used up to 0.5% offset yield and beyond that a speed of 0.05 inches per minute was used.

Strain measurements at elevated temperatures were approximated by recording cross-head motion since a suitable extensometer was not available.

At room temperature, a microformer type extensometer was used for strain measurements. This extensometer was calibrated using a precision micrometer once each day of testing.

In both cases, it was assumed that strain was uniform over the reduced section of the specimen.

6.2 Tests on Vascojet 90 Steel Sheet

6.2.1 Test Specimens

Specimens were machined from Vascojet 90 sheet supplied by AGARD. The scheme by which the test specimens were machined was that given by Supplement No.1 of February 1966 to the Refractory Metals Cooperative Testing Programme. The sheet orientation used was such that specimen No.1 contained the sheet marking 100. The specimen dimensions were those used for the TZM material with one exception. Because of the size of the blanks which resulted from the machining scheme used, it was necessary to reduce the overall length of the room temperature specimen from 4 inches to $3\frac{7}{8}$ inches. This change only affected the grip section and thus the gauge section remained identical to that previously used.

6.2.2 Description and Characteristics of Tensile Test Equipment and Methods.

The tensile test machine used was an Instron 10,000 pound capacity model with constant cross-head speed. In machines of this type, load is measured by a load cell to which the pull rod is directly attached. Calibration is by means of dead weights which are hung on the pull rods. The machine was calibrated once each day of testing.

Strain rate was controlled by cross-head motion for all tests. A cross-head speed of 0.005 inches per minute was used up to 0.5% offset yield and beyond that a speed of 0.05 inches per minute was used.

Strain measurements for the simulated elevated temperature tests were approximated by recording cross-head motion as had been done previously.

For the room temperature tests, a microformer type extensometer was used for strain measurements. This extensometer was calibrated using a precision micrometer once each day of testing.

In both cases, it was assumed that strain was uniform over the reduced section of the specimen.

These test conditions were identical to those used in the TZM test programme.

7. LABORATORY VII

7.1 Specimens

To conserve material, an attempt was made to produce specimen blanks from the first sheet of TZM, i.e., No.15 by guillotining a sandwich of TZM between sheets of mild steel. This resulted in considerable loss of material from edge cracking, characteristic of working at temperatures below the ductile-brittle temperature range. The second sheet No.13, was therefore supplied to enable all the tensile tests to be undertaken on material from one sheet.

Machined edges of the gauge length and transition portions were finished by polishing longitudinally with 00 emery.

7.2 Tensile Testing

All tests were conducted in a 10,000 lb. capacity, model TT-C Instron machine. Loads were measured by a load-cell complying with Grade A. BS 1610 (1964). Cross-head speed was held at 0.005 in./min. until a strain of 0.5% offset was reached, and then increased to 0.05 in./min. and maintained at that value until fracture occurred.

Provision of double pin-joint assemblies at each end of the test specimens minimized non-axial loading.

7.3 Room Temperature Tests

In the tests at room temperature, all the test pieces were fitted with double sided extensometers using electrical transducers, of accuracy 1" $\times 10^{-4}$, for strain measurement up to 0.5% offset. Beyond this value of strain, the cross-head speed was increased, and some indication of the strain was provided by recording the movement of the cross-head.

7.4 Elevated Temperature Tests

The apparatus for tests at 1050°C and 1450°C consisted of an evacuated stainless steel chamber, surrounded by a 12 in. long nichrome-wound furnace. A second furnace, 2 in. long and molybdenum wound, was mounted within the chamber surrounding the gauge length of the specimen. The ends of the specimen were considerably cooler than the gauge length and no measurable distortion of the loading holes occurred. The pumping system consisted of a 100 l/s diffusion pump backed by a two-stage rotary pump. In most tests, the vacuum was better than the specified vacuum of 1×10^{-4} mm of Hg but in several tests rose to 1.2×10^{-4}.

Values of strain were estimated from a chart recording of cross-head movement. Three Pt/Pt 13% Rh thermocouples, attached by Pt 13%RH wire and protected from direct radiation by 0.002 in. thick tantalum foil, provided temperature measurements over the 1 in. gauge length. A fourth thermocouple within the tantalum radiation shields of the small furnace acted as sensing element for a solid state temperature controller.

To faciliate "outgassing" of the chamber, furnace, etc., during heating to the test temperature, a procedure was adopted in which the apparatus was assembled, the outer furnace switched on, and the system left to "outgas" overnight at a temperature of 400 to 450°C. The following morning, the molybdenum furnace was switched on and the specimen brought to test temperature within 60 minutes in most tests (75 minutes for several 1450°C tests). After a 15 minute period for temperature stabilisation, the test was commenced. The temperature gradient was approximately 20 deg C in the 1050°C tests, and 30 to 50 deg C in the 1450°C tests. In all tests, the temperature at the middle of the gauge length was higher than at the ends of the gauge length.

Temperature variation during the greater part of the individual tests was ±5 deg C, but a variation of about 20 deg C was recorded just before fracture in several of the 1450°C tests; this was a consequence of the higher elongation values in the 1450°C series.

Rupture elongation values were obtained from measurements between small, jig-made, marks which defined the gauge length.

7.5 Recrystallization Temperature of TZM

Specimen blanks approximately ½" × ¼" were cut from the material remaining after manufacture of the tensile specimens. After degreasing in acetone, a specimen was placed on either side of the hot junction of a Pt/Pt 13% Rh thermocouple, covered with 0.002 in. thick tantalum foil and bound with molybdenum wire. The tantalum foil acted as a radiation shield and a protective getter for the TZM specimen. The complete assembly was mounted on a molybdenum/stainless steel rod which could move through a gas-tight seal at one end of a platinum wound furnace against a flow of 99.999% argon.

After remaining in a cool (100 to 150°C) part of the furnace for 10 minutes to permit purging of entrapped air, the specimens were passed into the hot zone where the temperature was monitored by a second thermocouple. Within 5 minutes the specimen reached test temperature. During the one hour soaking period, a temperature variation of ±2 deg C was recorded. The assembly was withdrawn to the cooler part of the furnace, and the temperature fell to 100 to 150°C within ten minutes.

Longitudinal and transverse sections of the specimens were prepared. After etching by swabbing with a solution of 10 g $K_3Fe(CN)_6$; 10 g NaOH; 80 ml water, an estimate of the amount of recrystallization was made using a magnification of 100.

8. LABORATORY VIII

8.1 Specimens

Blanks 20 mm wide and 77 mm long were cut with a hand saw made of a tool steel containing 12% Co. Shearing was not retained, because it resulted in severe cracking. The test pieces had the following dimensions:

total length	76.2 mm
gauge	25.4 mm
width at gauge length	6.35 mm
fillet radius	8.00 mm
pinhole diameter	8.00 mm
width at grip head	19.00 mm.

In comparison to the dimensions suggested in the AGARD document, the pin hole diameter was increased. Previous tests had in fact shown that the pin hole diameter should be higher than the gauge length in order to avoid distortion of the hole during the test. Furthermore the fillet radius was increased to 8 mm to insure a more progressive width variation.

The specimens were machined from the blanks by milling, using tungsten carbide tools.

Pin holes were first drilled with a calibrated fixture assuring a perfect reproducibility. The specimens were then machined by 5 on a fixture allowing the obtention of a good axiality of the holes relatively to the gauge length. The surface finish at the gauge length was the delivered surface one: no grinding, machining or etching was carried out. The edges of

the gauge length were carefully filed and polished longitudinally with 00 emery paper. The dimensions of the test pieces were carefully controlled. The maximum variation in the width of the gauge section in the same specimen was of ±0.01 mm. The minimum width was at the center of the gauge length.

8.2 Tensile Equipment

The tensile tests were carried out with an Instron tensile machine, type TT-C. Load was measured by means of high sensitivity strain gauge load cells, with an accuracy of better than ±0.5% for ranges extending from 100 to 5,000 kgs. The speed of the screw driven cross head varied from 0.005 to 5 cm/min.

For strain measurement the drum speed of the recorder was synchronous but independent of the cross head movement. The drum speed ranges from 0.2 to 50 cm/min.

Pull rods and pins were made of TZM molybdenum alloys. The threaded pins were held in place by means of screws. This system favoured the obtention of a good axiality.

The load cell was calibrated by means of 100 kg or 1 T dynamometer attached in the system instead of the specimen. This method allowed to take into account the friction between pull rods and the vacuum joints and also the effect of vacuum in the chamber. Thus the accuracy in load measurement exceeded ±0.5%.

For all tests a single cross-head speed of 0.5 mm/min. was used. This was constant with an accuracy of 1%. The axiality of the specimens relatively to the pulling rods was evaluated by means of comparators (1 micron sensitivity), mounted along the pull rods and the specimen. These measurements confirmed that the axiality of the system was correct.

Elongation measurements were carried out by means of markings 25.4 mm apart made with a tungsten carbide tip. The distance of the markings before and after test were measured with a sensitivity of 0.1 mm.

8.3 Method of Heating and Temperature Measurement

For elevated temperature tests a Brew furnace was attached on the Instron machine. The furnace has a tantalum - resistor fed through a voltage stabilizer-transformer. The voltage may vary from 0-15 V whereas the current may reach 800 A. For a given setting the voltage is stabilized with a precision of ±0.1%.

For temperature measurements at 1050°C and 1450°C PtRh 20% - PtRh 40% thermocouples were used, whereas at 1800°C WRe 5% - WRe 26% were used. The thermocouples were calibrated versus the melting point of pure substances:

Tin	231.9°C ± 0
Lead	329.4°C ± 0.0
Zinc	419 5°C ± 0.0
Silver	960.8°C ± 0.0
Gold	1063.0°C ± 0.0
Manganese	1244°C ± 3
Nickel	1453°C ± 0
Platinum	1769°C ± 0
Alumina	2045°C ± 5.

For all tests the specimens were brought to temperature in 30 minutes. Before loading the specimens were held at temperature for 15 minutes.

Preliminary tests were carried out with three thermocouples attached to the top, middle and bottom of the gauge length. The temperature variation along the gauge length was of ±1°C at 1050°C, ±2°C at 1450°C and ±4°C at 1800°C. During the actual tests one thermocouple was attached at the middle of the gauge length with an asbestos rope. The temperature variation during a test was of ±3°C at 1050°C, ±3°C at 1450°C and ±5°C at 1800°C.

For the tests at room temperature the latter was of 22 ±2°C.

8.4 Environment

All tests were carried out in vacuum obtained by means of an oil diffusion pump with a capacity of 750 L/sec. at 10^{-4} Torr and a backup rotary pump with a capacity of 8.7 m^3/hour. The enclosure in vacuum had a volume of 15.5 litres. A water cooled cold trap prevented any contamination of the furnace atmosphere by oil.

During the tests the degree of vacuum reach was as follows:

20°C	1×10^{-5} mm Hg
1050°C	2×10^{-5} mm Hg
1450°C	3×10^{-5} mm Hg
1800°C	4×10^{-5} mm Hg

8.5 Recrystallization Temperature

Specimens 10 mm wide and 20 mm long were subjected to 1 hour heat treatments at temperature between 1200°C and 1800°C, under vacuum in a tantalum resistor furnace (10^{-5} mm Hg). Heating time to temperature was of 25 min. and cooling time hours to 100°C was of 1 hour. During the treatments the temperature variation was of ±3°C.

9. LABORATORY IX

9.1 Specimens

The specimens used had a gauge length of 1 in. and a fillet radius of 12 mm. A special grip device was used to insure a better alignment of the load. The specimens were machined by precision milling. The gauge length edges were longitudinally polished with 00 emery paper.

9.2 Room Temperature Tests

A 10 T hydraulic Losenhausen machine was used. Load was recorded mechanically. Strain was determined by means of a 1 in. induction extensometer, attached to the specimen. Load was transmitted through self-aligning joints and auto-serrating grips.

9.3 Elevated Temperature Tests

For this purpose, a modified Heraeus vacuum was used. Loading was ensured through a motor-gear system with variable controlled speed. The cross-head speeds were chosen in order to conform to the specified strain rates.

For these tests a special self-aligning grip system was adopted.

Load was measured through an induction Vibrometer load cell. To avoid errors arising from the friction of the pull rods on the vacuum seals, the load cell was placed in the vacuum chamber. A cooling system prevented the heating of the load cell and vacuum seals. The accuracy of load measurement was better than 1% for load ranges of 100 and 500 kgs.

Strain was measured by means of extensometer arms attached to the ends of the specimen. The differential transformer extensometer was located in a cool place of the system and had a useful range of 7 mm and a precision of 1%.

A tantalum resistor, 50 mm in diameter and 300 mm long was used for heating the specimen. Test temperatures up to 1450°C were measured by means of Pt-PtRh thermocouples. At higher temperatures a disappearing filament monochromatic pyrometer was used.

Thermocouples attached to the specimen's gauge length showed that the variation along this length was lower than 0.5% of the nominal test temperature.

Heating time at temperature was of 20-30 min. The required temperature was maintained for 15 min. before loading.

The vacuum during all elevated temperature tests was of 10^{-4} Torr. The toom temperature in the laboratory was constant at 23°C ±2°C.

9.4 Recrystallization Tests

Specimens 10 mm wide and 20 mm long were heated to temperature in 20 - 30 min., in vacuum. Holding at temperature was of 1 hour. Cooling time was of 45 min. Vickers hardness was determined with a 500 gr. load.

10. LABORATORY X

10.1 Recrystallization Temperature

According to the specification of AGARD instruction MGRT 304, Appendix II, September 1964, the recrystallization temperature was determined by hardness measurements and metallographic examination.

The specimens (13 x 13 mm) were annealed in a vacuum furnace with tungsten heating elements. The pressure during the time to annealing temperature (40 min.) was below 5.10^{-5} Torr and at annealing temperature (1 hour) 2.10^{-5} Torr. The temperature has been measured simultaneously by means of a disappearing filament optical pyrometer and Pt/PtRh 10 thermocouples. The specimens were polished and etched using standard metallographic procedures.

10.2 Tensile Tests on TZM Sheet at Room and Elevated Temperatures

10.2.1 Test Specimens

The test specimen were of the same form and size at all testing temperatures. The blanks and the specimens were machined by milling and the edges of the gauge section were polished longitudinally with 00 emery, so that all remaining polishing scratches were parallel to the longitudinal axis of the specimen

10.2.2 Test Equipment

Tests were conducted with a Tinius Oelsen XY - Electomatic universal testing machine, into which a Marshall vacuum tensile furnace to operate at temperatures up to 2760°C was fitted One half of the circular-shaped heating element, fabricated as tungsten wire mesh, is mounted on the water-cooled front-opening door and the other half on the cold-wall furnace body

The load applied by the testing machine was transmitted to the specimen by a pin and hole assembly. The coaxiality of the loading was checked by strain measurement with extensometers and strain gauges, attached to opposing sides of the specimen. The maximum superimposed bending stresses were about 3% of the nominal tensile stress.

A newly developed and constructed extensometer, applicable up to testing temperatures of approximately 2500°C, records the specimen strain. The elongation between the two 3 mm diameter holes is measured with this device. The correlation between the nominal elastic strain between the two holes and that of the 1 in. gauge length is 0.82. The offset strain is the relationship of the recorded plastic elongation to the total length which experiences plastic deformation (approx. 30 mm).

The stress-strain and time-strain-diagrams were recorded simultaneously at all testing temperatures (room temperature, 1050°C, 1450°C and 1800°C). The strain rate increases at constant strain rate in accordance with the specification.

Temperature measurement was accomplished simultaneously be means of a disappearing filament pyrometer and Pt/PtRh 10 thermocouples up to 1600°C and WRe3/W Re 26 thermocouples beyond 1600°C; the thermocouples were fixed to the specimens by electric resistance spot welding.

The test conditions are summarized in the following section.

10.2.3 Test Conditions

(a) At room temperature.

Geometry of specimen: normal modified. Edge surface finish: deburring and 00 polishing.

Tensile equipment: Tinius Oelsen XY-Electomatic (6 T).

Loading system: electro-mechanical with adjustable, constant speed drive.

Testing Temperature °C	up to approx. 0.5% offset (kg)	Load range beyond approx. 0.5% offset (kg)
room	1200	1200
1050	600	600
1450	150	300
1800	60	150

Weighing system: electro-mechanical: weighing lever with torque bars and differential transformers.

Accuracy of load measurement: ½% of the applicable load range.

Atmosphere: Vacuum 10^{-2} Torr.

Strain measurement: Extensometer for elevated temperature (own design).

Accuracy of length measurement: 0.00125 mm.

Accuracy of strain measurement: 0.0025%.

Strain rate: 0.5% min. up to approx. 0.5% offset.
5% min. beoyond approx. 0.5% offset.

Cross-head speed: variable. Testing temperature: 22°C ±1°C.

(b) At elevated temperatures.

In addition to the conditions listed at a)

Heating element: tungsten wire mesh.

Time to testing temperature: 60 min: heating rate for a given testing temperature approximately constant.

Atmosphere: vacuum up to testing temperature 10^{-4} -2. 10^{-5} Torr. At testing temperature 2. 10^{-5} Torr.

Temperature measurement: 1050°C and 1450°C: Pt/PtRh10 thermocouples and optical pyrometer

1800°C: WRe3/WRe26 thermocouple and optical pyrometer.

Variation of temperature during test: ±2°C

Variation of temperature along gauge length: 1050°C ± 4.5°C
1450°C ± 3°C
1800°C ± 3°C.

10.3 Tensile Tests on Vascojet 90 (15CDV6) Steel

10.3.1 Test Conditions

Geometry of specimen: normal modified.

Edge surface finish: grinding and 00 polishing

Tensile equipment: Tinius Oelsen XY Electomatic (6 T)

Load range: 600 kg up to approx. 0.5% strain
1200 kg from approx. 0.5% strain

Accuracy of load measurement ±1/2% of the applicable load range

Strain rate: 0.5% min. up to 0.5%
5%/ min. from 0.5% up to rupture.

Strain measurement:

(a) Conventional tensile tests:

Extensometer (Tinius Oelsen).

Accuracy of the change of length measurement 1/400 mm.

Accuracy of strain measurement 0.01%.

For Young's modulus determination the results of the Tinius Oelsen.

Extensometer were checked by measurements with Huggenberger.

Extensometer: accuracy of the change of length measurement 10^{-4} mm
accuracy of strain measurement 0.0005%.

(b) Tensile tests under the conditions used for elevated temperature tests:

Extensometer for elevated temperatures (own construction).

Accuracy of strain measurement 0.0025%.

Room temperature 22 ± 2°C.

11. LABORATORY XI

11.1 Recrystallization Temperature Determination of TZM

Metallographic samples from the as-received TZM sheet number 8 material were annealed in a vacuum/induction heating unit which was pumped continuously to maintain the desired low pressure. The temperature range of interest was 1300°C (2372°F) to 1525°C (2777°F). Ten samples were exposed at 25°C (77°F) intervals up to 1525°C. The vacuum in the hot zone of the furnace varied from 8×10^{-6} to 3×10^{-6} mm Hg.

The samples were laid on a tungsten hearth plate 2" x 2". All temperatures were measured by means of a tungsten vs. tungsten -26% rhenium thermocouple located in the center of the hot zone and also by an Optrix disappearing filament pyrometer.

The samples were mounted in bakelite and electro polished in a Buehler 1721-1 AB Electro Polishing cell. The etching reagent used was Murakami's Reagent (10 gr sodium hydroxide, 10 gr potassium ferrocyanide, 100 ml distilled water). Samples were immersed for approximately 30 seconds. Metallographic microphotographs at 100 X were then taken.

Five Vicker diamond pyramid hardness readings were subsequently taken on each sample.

11.2 Room Temperature Tests on Vascojet 90

Ten room temperature tests were carried out in air using the same equipment, techniques and procedures as reported in section 11.3.1.for the TZM Sheet No.7 at room temperature. Constant head movement corresponding to 0.005 in./in./min. of strain was used instead of pacing strain. Five additional room temperature tests were performed using this same apparatus and procedures except that the specimens were held by pins instead of the V-grips.

Ten room temperature tests were conducted under the same vacuum conditions used for the elevated temperature tests. In this case, the same equipment and procedures, omitting the temperature measurements and heating method, were used as reported in Section 11.3.2. for the TZM at elevated temperatures.

11.3 Room and Elevated Temperature Tests on TZM Sheet No.7

11.3.1 Room Temperature

The tensile test machine used was an Instron Model TT-C. It has a constant cross-head speed and is equipped with a Leeds and Northrup X-Y recorder. In machines of this type, load is measured by a load cell which has a flexible self-aligning coupling. The load weighing accuracy is ±0.50% of indicated load or ±0.25% of recorder scale, whichever is greater, for all load ranges. The load cell was calibrated by applying chrome plated calibrating weights, procured from the Instron Company, to pull rods hung from the load cell. This calibration was checked whenever it was considered necessary throughout the test programme.

Specimen deformation was measured by a Baldwin-Lima-Hamilton (BLH) model B3M extensometer. This is a high magnification snap-on non-averaging extensometer. The extensometer was calibrated with a precision micrometer calibration device manufactured by Arizona Tool and Die Company. This calibrator had a least scale reading of 0.0001 inch with a vernier scale reading to 0.00002 inches. Therefore the strain system accuracy as determined by calibration was at least 0.0001 in./in. This calibration was also checked frequently during the test programme.

The strain rate was controlled using the Instron pacing equipment in conjunction with the variable cross-head speed control. All room temperature specimens were loaded at a strain rate of 0.005 in./in./min to approximately 0.010 inch deformation whereupon the

extensometer was removed and the strain rate was increased to 0.05 in./min. of head movement until failure.

Pin holes were machined into the specimens by eloxing (an electrical discharge method). Because of the rough surface developed by this machining method and the inherent low toughness of TZM at room temperature, the room temperature specimens failed initially at the pin holes rather than in the gauge section. To circumvent this problem room temperature gripping of the test specimens was accomplished by utilizing V-grips. Three 360^0 universal installed in the load train provided axiality of loading.

It was noted during the first couple of tests that failure of the specimens was accompanied by fragments shattering from the specimen. In order to facilitate retrieval of the fragments a collector was placed around the specimen after the extensometer was removed. With this apparatus it was possible to retrieve all pieces of the failed specimens so that accurate measurements of elongation could be made.

11.3.2 Elevated Temperature Tensile Equipment and Procedures

The elevated temperature tensile tests were performed in a vacuum furnace having tantalum heating elements (8 inches in length) which radiated heat to the specimen. The vacuum furnace was mounted on an Instron model TT-C-L tensile machine. This machine has characteristics similar to the one used for the room temperature tests. The load weighing accuracy and calibration of the load cell were the same as reported for the room temperature tests. The load applied by the testing machine was transmitted to the specimen by a pin and grip assembly.

Strain measurements at the elevated temperatures were obtained with a Riehle (RA-16774) extensometer. The output from the extensometer was fed through an Automatic Timing and Control (ATC) demodulator and recorded on a Varian X-Y recorder. The specimen deformation was measured by molybdenum attachments secured to the specimen. The molybdenum extension arms protruded through the bottom of the heat shield assembly and coupled to the extensometer. The accuracy of this strain system was determined in the same manner as the room temperature tests and was at least within 0.0001 in./in.

Because of the difficulty in pacing strain at elevated temperature the load was applied at constant head movement instead of at constant strain rate. The head movement was controlled at 0.005 in./min. to approximately 0.015 in. of deformation. At this point, the speed was increased to 0.05 in./min. until failure.

Temperature measurements were accomplished using chromel-P/alumel thermocouples at the 1050°C test temperature and using tungsten - 3% rhenium/tungsten - 26% rhenium thermocouples at the 1450°C and 1800°C test temperatures. The chromel P/alumel thermocouples were made from coils that were calibrated by the producer using a National Bureau of Standards No.27 platinum wire. The producer of the W 3%Re/W 26%Rh thermocouple wire supplied the following information: (a) The wire was a premium grade and 0.010 inch in diameter, (b) a laboratory standard Platinum vs. Platinum 10% Rhodium Thermocouple traceable to the National Bureau of Standards, Test #16951 was used to calibrate the sample wires to 1200°C. Above that point, an optical pyrometer traceable to the National Bureau of Standards Test #162016 was used. (c) EMF (mv) vs temperature (°C) was obtained from 400°C to 2300°C in 100°C increments.

The thermocouples were attached by wiring the thermocouple bead to the specimen surface. Alumina ceramic simulators were used on the thermocouples in the hot zone of the furnace. The Cr-Al thermocouples were connected to a Technique Inc. reference junction box and the W-3Re vs. W-26Re thermocouples were run to an ice bath reference junction and then to the temperature recorder controller. A Leeds and Northrup (L & N) Speedomax Type H temperaure recorder-controller was used to maintain the specimen temperature. This recorder-controller had a controlling accuracy of ¼ of one percent full scale range. The full

scale range was 25 mv. and each division on the indicating scale was 0.125 mv. An L & N temperature potentiometer with scale divisions of 0.1 mv was used as a secondary standard during each test.

Each thermocouple was used for only four tests at which time it was discarded. This represented approximately five hours of elevated temperature exposure. From previous testing it has been found that the calibration changes less than 5°C for the five hours of exposure.

In the heating system there was no means of controlling the temperature gradient along the gauge length of the specimen. Therefore the temperature distribution was monitored for only the first test at each temperature. Thereafter, the specimens were located within the heating system in the same relative position. The indicated temperature gradient was ±7°C at 1050°C, ±7°C at 1450°C and ±7°C at 1800°C. The indicated control thermocouple temperature variation during a test and from test to test was less than ±3°C for the 1050°C tests and ±5°C for the 1450°C and 1800°C tests.

To maintain a vacuum environment of better than 10^{-4} mm of mercury during heat-up, the heating rate was not constant since outgassing of the furnace was not constant. The heating time at temperature for the majority of the specimens at each temperature are given in the following table:

Test Temperature	Heating Time minutes	Time at temperature minutes
1050	20	15
1450	25	40
1800	30	20

At the 1450°C test temperature, the summation of heating time and time at temperature was held closely to 60 minutes. This time period was recommended in the AGARD progress report number 1, November 1965.

The environment at the elevated testing temperature was 6×10^{-5} mm of mercury or better. A vacuum diffusion pump was used to obtain this environment. A hot filament ionization gauge, NRC Equipment Corp. type 507, in conjunction with a NRC type 710 thermocouple-ionization control was used to measure the vacuum pressure.

11.4 Room and Elevated Temperature Tests on TZM Sheet Number 8

11.4.1 Room Temperature Tensile Test Equipment and Procedures

All room temperature apparatus and procedures were the same as described in section 11.3.1 for sheet number 7 except that the tensile load was applied by pins instead of the V-grip loading technique. Axiality of loading was checked by placing strain gauges on two specimens and loading one specimen in the tensile machine. Since no variation in the strain readings were observed by rotating the active specimen 180° relative to the grips, axiality was assumed.

11.4.2 Elevated Temperature Tensile Test Equipment and Procedures

The elevated temperature tensile tests on this sheet were performed in an electron beam heating vacuum chamber. Load was applied by the Instron model TT-C tensile machine that was described in section 11.3.1 for the TZM sheet number 7 at room temperature.

Elevated temperature was measured and controlled using thermocouples that were made from producer calibrated coils. The thermocouples used were: chromel-P/alumel at 1050°C, platinum/platinum 10% rhenium at 1450°C, and tungsten - 3% rhenium/tungsten -26% rhenium at 1800°C.

Three means of measuring strain at elevated temperature were attempted using specimens from sheet number 8. These were (1) electro-optical extensometer (2) photographic optical system, and (3) cross-head movement.

The electro-optical extensometer was designed to optically track the mechanical movement of two target projections attached to the ends of the gauge length of a specimen. The advantage of such a system in applications such as elevated temperature tensile strain measurements is that the sensing device is outside the vacuum furnace and free from the effects of the extreme test environments. However, since measurement is performed optically the following items must be considered: target quality, target illumination and background proper lens for target range, and effects of environment chamber interior/conditions and observation ports on the target image seen by the extensometer.

Under somewhat ideal conditions, i.e., room temperature, black non-reflecting projections, diffused lighted background, targets outside any type chamber, and no mechanical vibrations; the electro-optical extensometer is a reliable and accurate instrument. Numerous tests were performed with this extensometer with specimens inside the vacuum chamber using different types of lighting. The results of these tests show that further work must be done to improve the set-up and arrangement of the testing equipment.

Since further work on the electro-optical extensometer was required a photographic optical means employing a 70 mm camera was used to measure the displacement of tabs spot welded on the specimens. During these tests, the cross-head movement of the tensile machine was also recorded. Each time a photograph was taken, the load versus head movement chart was automatically marked.

APPENDIX F

Analysis of Variance Computations

INTRODUCTION

A more quantitative assessment of the variability sources in the results of the programme considered is provided by the analysis of variance computations. The results of these computations are reproduced in Table 8-15 and 17-32 numbered respectively on the basis of the presentation in the main part of this report. The intralaboratory (or residual) mean square provides an estimate of the experimental error variance σ_0^2, whereas the interlaboratory mean square provides an estimate of $\sigma_0^2 + n\sigma_1^2$ where σ_1^2 is the variance due to interlaboratory variability and n is the number of tests in each laboratory.

Although the chance causes resulting in the experimental error may well not be the same for all the laboratories, the computation of the intralaboratory mean square provides a fair estimate of the average experimental error variance based on a large number of degrees of freedom. In the present programme the estimate of σ_0^2 might be to some extent biassed because of the fact that the samples distributed to the participating laboratories were not completely randomized. However, the possible bias arising from non-homogeneous samples is probably negligible in view of the considerations given in Appendix C. The analysis of variance tables reproduces also the ratio of the interlaboratory mean square to the intralaboratory one. The values of F at the 99% significance level are also reproduced for the corresponding degrees of freedom. The F-test actually checks the hypothesis that $\sigma_1^2 = 0$, i.e. that the interlaboratory variability is not significant.

In the case that the F-test shows that the mean values differs significantly it is usual to subject the mean values to a more thorough examination. Several methods are available for this purpose, and in the present case only a simple method will be considered. This consists in determining the least significant difference which the data would demonstrate to be statistically significant[1,2]. The comparison of the differences of means to the least significant difference enables one to determine the groups of means which statistically cannot be distinguished from each other at a specified level of significance. The least significant difference (LSD) is given by the relation:

$$LSD = t_\alpha S\sqrt{\frac{2}{n}}$$

where: S = is the square root of the intralaboratory mean square

n = number of tests used in the determination each mean. In this case, n is generally equal to 10.

t_α = the parameter corresponding to the 100 $(1-2\alpha)$% level of significance and to the degrees of freedom used in the determination of S.

The values of LSD as computed from the above relation for the 95% and 99% significance level are also reproduced below the analysis of variance tables. The value of n was of 10 although, for some tests and for some laboratories, the number of repetitive tests was lower than 10. Finally the tables reproduce the groups of laboratories the mean values of which cannot be distinguished at the 95% or the 99% significance level.

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[1] VILLARS, D.S. — Statistical Design and Analysis of Experiments for Development Research, WM.C.Brown Cy, Dubuque, Iowa, 1951.

[2] DAVIES, O.L. — Statistical Methods in Research and Production, Oliver and Boyd, London, 1967.

Discussion of the Results

The examination of the results of the F-tests shows that for both materials considered and almost for all tests the interlaboratory variability is highly significant. The exception are the following:

- Vascojet conventional modulus (Table 11, 3 Labs.)
- Vascojet high temperature modulus (Table 15, 3 Labs.)
- TZM RT modulus (Table 20, 4 Labs.)
- TZM 1450 modulus (Table 28, 2 Labs.)
- TZM 1800 modulus (Table 32, 2 Labs.)

The fact that all the exceptions relate to modulus determination is remarkable. Indeed all modulus determinations, except that at 1050°C (Table 24), resulted in non-significant interlaboratory variations. Although the number of laboratories involved was generally small, the fact that modulus determinations give rise to good interlaboratory reproductibility may be due to the close control of operating conditions required for these tests and also to the fact that modulus is a structure-insensitive property. The computation of the least significant difference allows, as stated above, the identification of the laboratories whose mean values cannot be distinguished at the specified level of significance. As shown in the Tables the number of distinguishable groups varies depending on the extent of the interlaboratory variability. The determination of these groups is, perhaps, more informative than the control charts given in the main part of this report, although the latter has the advantage of comparing the individual laboratory mean to the grand mean. In addition, the determination of the control charts is, to some extent, easier.

Considering the large number of factors which might affect the final results it is impossible to assign to different groups of statistically equivalent laboratories a specified cause of variation.

TABLE 8

Vasco Conv. UTS

Results: All, except Laboratories:

Source	Sum of Squares	D.F.	M.S.	F	F 99(8,81) = 2.74
Interlaboratory	168.551	8	21.06	30.75	
Intralaboratory	55.500	81	0.68		
Total	224.051	89	2.51		

Significance Level: 95%

LSD = 0.740
I, XI
XI, VIII
VIII, II
VII, X, V
III
VI

Significance Level: 99%

LSD = 0.977
I, XI, VIII
VIII, II
II, VII, X, V
III, VI

TABLE 9

Vasco Conv. YS

Results: All, except Laboratories:

Source	Sum of Squares	D.F.	M.S.	F	F 99(8,81) = 2.74
Interlaboratory	34.962	8	4.37	6.025	
Intralaboratory	58.750	81	0.72		
Total	93.712	89	1.05		

Significance Level: 95%

LSD = 0.761
I, XI
VIII, II, VII, V, III, VI, X

Significance Level: 99%

LSD = 1.007
I, IX
VIII, II, VII, V, III, VI, X

TABLE 10

Vasco Conv. El

Results: All, except Laboratories:

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(8,81) = 2.74*
Interlaboratory	128.833	8	16.10	10.3	
Intralaboratory	126.578	81	1.56		
Total	255.411	89	2.86		

Significance Level: 95%

LSD = 1.118
VI, XI
XI, VIII, III
VIII, III, X
X, II, I
II, I, VII, V

Significance Level: 99%

LSD = 1.475
VI, XI, VIII
XI, VIII, III, X
VIII, III, X, II
III, I, X, II
II, I, VII, V

TABLE 11

Vasco Conv. Mod

Results: All except Laboratories:

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(2,27) = 5.49* *F 95(2,27) 3.35*
Interlaboratory	7.442	2	3.72	3.87	
Intralaboratory	25.939	27	0.96		
Total	33.382	29	1.15		

Significance Level: 95%

LSD = 0.876
II, X
X, XI

Significance Level: 99%

LSD = 1.385
II, X, XI

TABLE 12

Vasco HT UTS

Results: All, except Laboratories

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,90) = 2.6*
Interlaboratory	329.155	9	36.57	53.25	
Intralaboratory	61.812	90	0.68		
Total	390.967	99	3.94		

Significance Level: 95%

LSD = 0.741
- VIII
- II, VII, X, I
- III, XI, V
- VI
- IV

Significance Level: 99%

LSD = 0.980
- VIII
- II, VII, X, I
- I, III
- III, XI, V
- VI
- IV

TABLE 13

Vasco HT YS

Results: All, except Laboratories:

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,88) = 2.64*
Interlaboratory	183.459	9	20.38	26.1	
Intralaboratory	68.718	88	0.78		
Total	252.178	97	2.59		

Significance Level: 95%

LSD = 0.790
- I, VIII
- VIII, VII
- VII, III, II, V
- II, V, X
- V, X, XI
- X, XI, VI
- IV

Significance Level: 99%

LSD = 1.04
- I, VIII, VII
- VIII, VII, III
- VII, III, II, V
- II, X, XI
- X, XI, VI
- IV

TABLE 14

Vasco HT El

Results: All, except Laboratories:

Source	Sum of Squares	D.F.	M.S.	F	F 99(9,89) = 2.64
Interlaboratory	145.040	9	16.11	11.69	
Intralaboratory	122.687	89	1.37		
Total	267.728	98	2.73		

Significance Level: 95%

LSD = 1.050
VI, IV, XI
XI, VIII, X
VIII, X, III
X, III, V
III, V, I
V, I, VII, II

Significance Level: 99%

LSD = 1.38
VI, IV, XI, VIII
IV, XI, VIII, X
VIII, X, III, V
X, III, V, I
III, V, I, VII, II

TABLE 15

Vasco HT Mod

Results: All, except Laboratories:

Source	Sum of Squares	D.F.	M.S.	F	F 99(2,26) = 5.53 F 95(2,26)3.37
Interlaboratory	11.159	2	5.57	5.23	
Intralaboratory	27.714	26	1.06		
Total	38.874	28	1.38		

Significance Level: 95%

LSD = 0.923
IV, X
XI

Significance Level: 99%

LSD =1.28
IV, X
XI

TABLE 16

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TABLE 17

TZM RT UTS

Results: All, except Laboratories:
Labs III, VI and XI (e)

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,83) = 2.64*
Interlaboratory	632.091	9	70.23	15.02	
Intralaboratory	388.125	83	4.67		
Total	1020.216	92	11.08		

Significance Level: 95%

LSD = 1.934
- I
- XI, IIa
- IIa, V
- V, IIb, V, X
- IIb, V, X, VIII, IV
- X, VIII, IV, IX

Significance Level: 99%

LSD = 2.55
- I, XI
- XI, IIa
- IIa, VII, IIb, V
- VII, IIb, V, X, VIII, IV,
- IIb, V, X, VIII, IV, IX

TABLE 18

TZM RT YS

Results: All, except Laboratories:
Lab III, VI and XI(e)

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,82) = 2.64*
Interlaboratory	984.272	9	109.36	35.03	
Intralaboratory	256.000	82	3.12		
Total	2240.272	91	13.62		

Significance Level: 95%

LSD = 1.58
- XI, I
- V, VII, X, IIb, IIa, IX
- VIII
- IX

Significance Level: 99%

LSD = 2,083
- XI, I
- I, V, VII, X
- V, VII, X, IIb, IIa, IX
- VIII
- IX

TABLE 19

TZM RT El

Results: All, except Laboratories:
Lab.III, IV, VI, XI(e)

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(8,72) = 2,77*
Interlaboratory	193.415	8	24.17	6.09	
Intralaboratory	285.648	72	3.96		
Total	479.063	80	5.98		

Significance Level: 95%

LSD = 1.781
- XI, I, X
- X, IX
- IX, IIb, V, VIII, VII
- IIb, V, VIII, VII IIa

Significance Level: 99%

LSD = 2.35
- I, X, IX
- X, IX, IIb
- IX, IIb, V, VIII, VII
- IIb, V, VIII, VII, IIa

TABLE 20

TZM RT Modulus

Results: All, except Laboratories:
Lab.VI and XIe

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(3,36) = 4.38*
Interlaboratory	5.375	3	1.79		
Intralaboratory	173.726	36	4.82		
Total	179.101	39	4.59		

Significance Level: 95%

LSD = 1.964
XI, IV, II, X

Significance Level: 99%

LSD = 2.65
XI, IV, II, X

TABLE 21

TZM 1050 UTS

Results: All, except Laboratories:
Lab.VI, XI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,79) = 2.64*
Interlaboratory	414.223	9	46.02		
Intralaboratory	219.000	79	2.77		
Total	633.223	88	7.19		

Significance Level: 95%

LSD = 1.489
IV, II, I
II, I, III, XI, V
III, XI, V, VIII
V, VIII, X
VII, IX

Significance Level: 99%

LSD = 1.97
IV, II, I, III, XI
II, I, III, XI, V
III, XI, V, VIII, X
VII, IX

TABLE 22

TZM 1050 YS

Results: All, except Laboratories:
Lab. VI and XI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(9,77) = 2.64*
Interlaboratory	841.698	9	93.52	27.16	
Intralaboratory	265.093	77	3.44		
Total	1106.792	86	12.86		

Significance Level: 95%

LSD = 1.659
II, I, IX
III, V, XI
XI, IV
IV, X, VIII
VII

Significance Level: 99%

LSD = 2.19
II, I, IX
IX, III
III, V, XI
V, XI, IV
XI, IV, X, VIII
VII

TABLE 23

TZM 1050 E1

Results: All, except Laboratories:
Lab. IV and XI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(8,70) = 2.77*
Interlaboratory	28.134	8	3.51	3.36	
Intralaboratory	73.229	70	1.04		
Total	101.364	78	1.29		

Significance Level: 95%

LSD = 0.914
IX, XI, X
XI, X, VII, V, III
X, VII, V, III, VIII
VII, V, III, VIII, II
V, VIII, III, II, I

Significance Level: 99%

LSD = 1.21
IX, XI, X, VII, V, III
XI, X, VII, V, III, VIII
X, VII, V, III, VIII, II
VII, V, III, VIII, II, I

TABLE 24

TZM 1050 Mod

Results: All, except Laboratories:

Source	Sum of Squares	D.F.	M.S.	F	F 99(2,25) = 5.57
Interlaboratory	33.637	2	16.81	9.12	
Intralaboratory	46.078	25	1.84		
Total	79.719	27	2.95		

Significance Level: 95%

LSD = 1.214
IV, XI
X

Significance Level: 99%

LSD = 1,69
IV, XI
XI, X

TABLE 25

TZM 1450 UTS

Results: All, except Laboratories:
Lab.IV, VI and XI

Source	Sum of Squares	D.F.	M.S.	F	F 99(8,71) = 2.77
Interlaboratory	313.396	8	39.17	30.04	
Intralaboratory	92.583	71	1.30		
Total	405.980	79	5.13		

Significance Level: 95%

LSD = 1.021
I
VIII, XI, V, II, IX
V, II, IX, X
II, IX, X, VII
III

Significance Level: 99%

LSD = 1.35
I
VIII, XI, V, II, IX, X
V, II, IX, X, VII
IV

TABLE 26

TZM 1450 YS

Results: All, except Laboratories:

Lab. IV and VI

Source	Sum of Squares	D.F.	M.S.	F	F 99(8,70) = 2.77
Interlaboratory	216.463	8	27.05	26.7	
Intralaboratory	70.944	70	1.01		
Total	287.407	78	3.68		

Significance Level: 95%

LSD = 0.900
I, V, II
V, II, XI
X, VIII, VII, IX
III

Significance Level: 99%

LSD = 1.19
I, V, II
V, II, XI
X, VIII, VII, IX
III

TABLE 27

TZM 1450 El

Results: All, except Laboratories:
Lab. IV, VII and XI

Source	Sum of Squares	D.F.	M.S.	F	F 99(8,70) = 2.77
Interlaboratory	3471.304	8	433.91	20.02	
Intralaboratory	1517.218	70	21.67		
Total	4988.523	78	63.95		

Significance Level: 95%

LSD = 4.164
III, VII
V, VIII, I, II
XI, IX, X

Significance Level: 99%

LSD = 6.13
III, VII
V, VIII, I, II
II, XI
XI, IX, X

TABLE 28

TZM 1450 Mod

Results: All, except Laboratories:
Lab. IV

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(1,16) = 8.53* *F 95(1,16) 4.49*
Interlaboratory	7.832	1	7.83	5.98	
Intralaboratory	20.968	16	1.31		
Total	28.801	17	1.69		

Significance Level: 95%

LSD = 1.023
XI
X

Significance Level: 99%

LSD = 1.49
XI, X

TABLE 29

TZM 1800 UTS

Results: All, except Laboratories:
Lab. VI and XI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(5,45) = 3.45*
Interlaboratory	40.918	5	8.18	34.06	
Intralaboratory	10.811	45	0.24		
Total	51.730	50	1.03		

Significance Level: 95%

LSD = 0.438
X
XI, IX
IX, II, III
VIII

Significance Level: 99%

LSD = 0.598
X
XI, IX, II, III
VIII

TABLE 30

TZM 1800 YS

Results: All, except Laboratories:
Lab. VI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(5,45) = 3.45*
Interlaboratory	84.157	5	16.83	212.13	
Intralaboratory	3.570	45	0.07		
Total	87.727	50	1.75		

Significance Level: 95%

LSD = 0.251
X, XI
II,
III
IX
VIII

Significance Level: 99%

LSD = 0.34
X, XI
XI, II
III
IX
VIII

TABLE 31

TZM 1800 El

Results: All, except Laboratories:
Labs. VI and XI

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(5,43) = 3.47*
Interlaboratory	10063.541	5	2012.70	14.67	
Intralaboratory	5899, 375	43	137.19		
Total	159[illegible].916	48	332.56		

Significance Level: 95%

LSD = 10.476
II, XI, IX, VIII
XI, IX, VIII, III
X

Significance Level: 99%

LSD = 14.15
II, XI, IX, VIII
X

TABLE 32

TZM 1800 Mod

Results: All, except Laboratories:

Source	*Sum of Squares*	*D.F.*	*M.S.*	*F*	*F 99(1,13) = 9.07* *F 95(1,13) 4.67*
Interlaboratory	3.880	1	3.88	4.52	
Intralaboratory	11.169	13	0.85		
Total	15.050	14	1.07		

Significance Level: 95%

LSD = 0.829
X
XI

Significance Level: 99%

LSD = 1.24
X, XI

APPENDIX G

Influence of Measurement Inaccuracies on Tensile and Creep Test Data

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1. INTRODUCTION AND SCOPE

The accuracy of the measurement of load, length, time and temperature for tensile and creep testing has been standardized rather completely (DIN, ASTM, British Standards etc.). The errors allowed in these measurements determine the accuracy of the data to be obtained, depending upon the behaviour of the material under consideration. The resultant error of the data is of interest and should be known to the designer.

The standards for correct measurement are based on extensive experience with common materials. The standardized testing of such materials and of those with similar properties leads therefore generally to small errors for the resultant data. The specifications however do not take sufficiently into consideration the influence of the rigidity of the testing machine, which also depends on the elasticity of clamping devices and the ends of the test specimens, on the strain rate[1].

Better methods and equipments for measurements in tensile and creep testing at temperatures beyond 1200°C are under development at present[2,3]. Specifications concerning the accuracy of measuring under such conditions already exist, but they cannot be realized in many cases. Furthermore several institutions have agreed upon procedures based on the accuracy made possible by well known methods and facilities, as has been done by AGARD for the "Cooperative Programme on Mechanical Testing of Refractory Metals".

This approach is advantageous for the explanation of the scatter in the resultant data from different laboratories caused by inaccurate measurements of individual factors of influence. The knowledge of the degree of precision is also useful for the evaluation of the different procedures applied by these laboratories and for the establishment of specifications more suitable to acceptance or collaborative testing, as in the "Round Robin Creep Testing Programme" planned by the Structures and Materials Panel, AGARD. A decision should also be made for the degree of accuracy to be required for the measurement of factors involved in tensile and creep testing for a given resultant inaccuracy of data. For example, the error caused by measuring the strain between shoulders at the ends of the gauge length instead of by extensometers attached with knife edges to a gauge length of constant cross-section, can be adequately eliminated in tensile tests[2], where as the strain in creep testing is determined precisely enough only by the latter method[4].

In the following sections the influence of measurement errors in tensile and creep testing on the precision of data will be discussed. By means of a nomogram the resultant error of data depending on inaccurately measured strain, temperature and strain rate can be determined. For different groups of materials the contribution of partial error to the overall inaccuracy for yield strength ultimate tensile stress and creep or rupture strength will be shown.

2. FACTORS OF INFLUENCE

In tensile testing, length load, temperature and time are measured. The total elongation and reduction of area are measured at room temperature and errors relating to these will

therefore not be treated here. The error in stresses induced by inaccuracies in the determination of the factors mentioned above will be considered. The relative error for the nominal stress is derived from the equation $\sigma = \frac{P}{F_0}$.

$$\boxed{\left|\frac{\delta\sigma}{\sigma}\right| = \left|\frac{\delta P}{P}\right| + \left|\frac{\delta F_0}{F_0}\right| = \left|\frac{\delta P_M}{P}\right| + \left|\frac{\delta P_E}{P}\right| + \left|\frac{\delta F_0}{F_0}\right|} \qquad \text{(I)}$$

where -

σ = nominal stress

$\delta\sigma$ = absolute error in nominal stress

P = load

δP = absolute error in load

$\frac{\delta P_M}{P}$ = relative error of measuring the load

$\frac{\delta P_E}{P}$ = relative error in load, caused by errors of measuring the strain ϵ, temperature T and strain rate $\dot{\epsilon}$ *)

F_0 = original cross section

δF_0 = absolute error in cross section

The tensile load depends on ϵ, T, $\dot{\epsilon}$; the error in measuring the area of the original cross section of the gauge length may be neglected. Therefore the error in the nominal stress resulting from the relative errors in $\frac{\delta\epsilon}{\epsilon}$, $\frac{\delta T}{T}$, $\frac{\delta\dot{\epsilon}}{\dot{\epsilon}}$ and dimensionless factors is given by the following equation:

$$\boxed{\left|\frac{\delta\sigma}{\sigma}\right| = \left|\frac{\delta\sigma_M}{\sigma}\right| + \left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| \cdot \left|\frac{\delta\epsilon}{\epsilon}\right| + \left|\frac{\partial\sigma/\partial T}{\sigma/T}\right| \cdot \left|\frac{\delta T}{T}\right| + \left|\frac{\partial\sigma/\partial\dot{\epsilon}}{\sigma/\dot{\epsilon}}\right| \cdot \left|\frac{\partial\dot{\epsilon}}{\dot{\epsilon}}\right|} \qquad \text{(II)}$$

The factors $\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right|$, $\left|\frac{\partial\sigma/\partial T}{\sigma/T}\right|$ and $\left|\frac{\partial\sigma/\partial\dot{\epsilon}}{\sigma/\dot{\epsilon}}\right|$ are called modulus of error in strain, temperature or strain rate; $|\delta\sigma_M/\sigma|$ is the relative error in nominal stress caused by load measurement inaccuracy.

In the linear elastic range of the stress-strain curve $\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| = 1$ and $\left|\frac{\partial\sigma/\partial\dot{\epsilon}}{\sigma/\dot{\epsilon}}\right| \simeq 0$; therefore, the error in the modulus of elasticity can be determined, if its temperature dependence and the precision of measuring the load, strain and temperature are known. Instead of the error in the yield strength the corresponding error in stress (equation II) may be taken for simplification.

In the following paragraph it will be shown that, although $\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| = 0$ at the maximum

*) the true strain ϵ_ω is equal to $ln(1+\epsilon)$.

of the nominal stress-strain curve, an error in strain influences indirectly the precision of determining the ultimate tensile stress due to the introduction of strain rate. For a realization of constant strain rate, which is advantageous for tensile tests in many aspects, the control of the speed of the cross-head corresponding to the time dependent output of the extensometer must be exact. Then an error in the strain rate is given only by the accuracy of measuring strain and time. A momentary deviation from a prescribed strain rate cannot change appreciably the rate of load and elongation, since the control mechanism reacts rather slowly. In many cases such deviation can be corrected for the evaluation of a recorded stress-strain or load-elongation diagram. For simplification the evaluation is based on the strain rate average $\dot{\epsilon}_m = \frac{\epsilon}{t}$. The relative error becomes

$$\boxed{\left|\frac{\delta\dot{\epsilon}_m}{\dot{\epsilon}_m}\right| = \left|\frac{\delta\dot{\epsilon}_R}{\dot{\epsilon}}\right| + \left|\frac{\delta\epsilon}{\epsilon}\right| + \left|\frac{\delta t}{t}\right|}, \qquad \text{(III)}$$

where the absolute errors are

$\delta\dot{\epsilon}_m$ average strain rate

$\delta\dot{\epsilon}_R$ control inaccuracy

$\delta\epsilon$ strain measurement

δt time.

This equation is also valid for the instantaneous strain rate, if the relative error in time and strain as basis for determining the strain rate, are considered. Attention should be paid to the direct influence of strain error on the error in stress in comparison with the influence of strain rate error.

With constant cross-head speed in tensile tests the strain rate increases generally several hundred per cent during the transition from elastic to elastic-plastic strain. This is principally due to the deformation of the testing machine and the ends of the test specimen. When a deviation of such an extent occurs the methods developed here allow only an estimation of errors in data.

The error in creep test data can also be calculated with equation II. When the errors from measuring load, cross-section area and time are neglected, the accuracy of creep limit is given by the following equation

$$\boxed{\left|\frac{\delta\sigma}{\sigma}\right| = \left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| \cdot \left|\frac{\delta\epsilon}{\epsilon}\right| + \left|\frac{\partial\sigma/\partial T}{\sigma/T}\right| \cdot \left|\frac{\delta T}{T}\right|} \qquad \text{(IV)}$$

The modulus of error in strain $\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right|$ cannot be determined directly from the creep-strain-time-diagram; therefore at specific times e.g. 1 h, 10 h, 100 h etc., the total strain and corresponding stress are taken from creep curves; these data points result in stress-strain curves with time as parameter, from which the modulus of error in strain can be determined. This modulus may be derived from stresses and related strains at different times and temperatures, when creep limits at different strains are given. The accuracy of determining the rupture strength is not influenced by a strain error.

3. NOMOGRAM FOR THE EVALUATION OF THE RESULTANT ERROR IN STRESS VALUES

A nomogram (Fig. 1) has been devised that demonstrates clearly the relations expressed by the equations II and IV as well as the influence of the individual errors. The entire nomogram is a composition of the nomograms I, II, and III for the individual errors due to measurement inaccuracies of strain, temperature and strain rate, depending on the moduli of error. With logarithmic coordinates, the curves for equal error are straight lines with a slope of 45°, for example:

$$\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| \cdot \left|\frac{\delta\epsilon}{\epsilon}\right| = \text{const} \longrightarrow \lg\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right| = \lg\text{ const} - \lg\left|\frac{\delta\epsilon}{\epsilon}\right| .$$

In the individual nomogram IV and V, respectively the errors $\left|\frac{\delta\sigma}{\sigma}\right|_\epsilon$, $\left|\frac{\delta\sigma}{\sigma}\right|_T$ and $\left|\frac{\delta\sigma}{\sigma}\right|_{\epsilon,T}$, $\left|\frac{\delta\sigma}{\sigma}\right|_{\dot\epsilon}$ are summarized. As an example, the resultant error that will be caused by the assumed errors

$\left|\frac{\partial\epsilon}{\epsilon}\right|$ - when ϵ is determined by measuring the cross-head movement - , $\left|\frac{\delta T}{T}\right|$ and $\left|\frac{\delta\dot\epsilon}{\dot\epsilon}\right|$

is illustrated for $\sigma_{0,2}$ of the molybdenum alloy TZM at 1800°C. The nomogram is useful particularly, if a given accuracy of the stress value is prescribed. Then the allowable individual errors $\left|\frac{\delta\sigma}{\sigma}\right|_\epsilon$, $\left|\frac{\delta\sigma}{\sigma}\right|_T$ and $\left|\frac{\delta\sigma}{\sigma}\right|_{\dot\epsilon}$ can be adjusted to the available means for measurement or control respectively.

4. MODULI OF STRESS DATA

The use of the nomogram requires the modulus of error in strain, temperature and strain rate to be known. In Figure 2 these moduli, for the $\sigma_{0,2}$ limit, are plotted versus temperature for some light metal alloys, superalloys, steels, and refractory metals. These data were determined on the basis of the total deformation corresponding to 0.2% yield strength.

For the tested materials, modulus of error in strain depends somewhat on temperature. Normally it cannot exceed 1 and the average value is about 0.2. The individual error in $\sigma_{0,2}$ from strain measurement inaccuracy is at most equal to the relative error in strain $\left|\frac{\delta\epsilon}{\epsilon}\right|$, but on an average not larger than $1/5\left|\frac{\delta\epsilon}{\epsilon}\right|$. Hereby the influence of the strain rate is not considered.

The modulus of error in temperature increases remarkably with rising temperature in some cases and sometimes exceeds 10. The individual error in the 0.2% offset yield strength due to inaccurate determination of the temperature, may be one order of magnitude higher than the relative error in temperature $\left|\frac{\delta T}{T}\right|$. Such a high modulus of error in temperature occurs not only in a range of temperature with a high $\partial\sigma/\partial T$, but also at much higher temperatures, since the ratio σ/T becomes smaller than $\partial\sigma/\partial T$. The average error in temperature amounts nearly to 3. This means that the individual error in the

$\sigma_{0,2}$ limit from temperature error becomes on an average three times the relative error in temperature $\frac{\delta T}{T}$.

The few data available on the modulus of error in strain rate indicate that this modulus of error clearly depends on the strain rate and temperature. The value of one seems to be seldom exceeded when a stress limit for a permanent set is determined with normal strain rate; the average will be about 0.2, as has been found for the modulus of error in strain.

For an average dependance of strain, temperature and strain rate the relative error in $\sigma_{0,2}$ can be roughly estimated as

$$\boxed{\left|\frac{\delta\sigma}{\sigma}\right|_{\sigma_{0,2}} \approx \frac{1}{5}\left|\frac{\delta\epsilon}{\epsilon}\right| + 3\left|\frac{\delta T}{T}\right| + \frac{1}{5}\left|\frac{\delta\dot{\epsilon}}{\dot{\epsilon}}\right|} \qquad \text{(Va)}$$

When the control of cross-head movement allows a nearly constant strain rate to be realized, consideration of equation III gives

$$\boxed{\left|\frac{\delta\sigma}{\sigma}\right|_{\sigma_{0,2}} \approx \frac{1}{2}\left|\frac{\delta\epsilon}{\epsilon}\right| + 3\left|\frac{\delta T}{T}\right|} \qquad \text{(Vb)}$$

The modulus of error in creep limits is sometimes much larger than the modulus of error in yield strength determined in tensile tests. Figure 3 shows that for an aluminum alloy this difference is remarkable. $\left|\frac{\partial\sigma/\partial\epsilon}{\sigma/\epsilon}\right|_{\sigma_{0,2}}$ increases rapidly with time, for which the creep limit is measured and becomes for 1000 h nearly sixteen times the error modulus measured in a tensile test. Therefore the measurement of strain in creep testing should be generally more accurate[4] than in tensile testing[2]. But in tensile testing the strain error $\left|\frac{\delta\epsilon}{\epsilon}\right|$ influences also the strain rate error, and therefore intensifies the influence of $\left|\frac{\delta\epsilon}{\epsilon}\right|$ on the resultant error in characteristic data due to the obvious dependance of yield strength on strain rate (see equations III, IV, and V).

5. SUMMARY

The design of structures must be based on the knowledge of the error in mechanical properties for the materials. The relative resultant stress error is given as a function of the relative error in factors of influence (strain, temperature, strain rate) and of dimensionless moduli of error which depend on the material behaviour. Reference is made to the effect of strain error on the strain rate error. The effects of various errors can be evaluated easily from a nomogram. The modulus of factors of influence are given for the yield and creep strength for several groups of materials and compared one with another.

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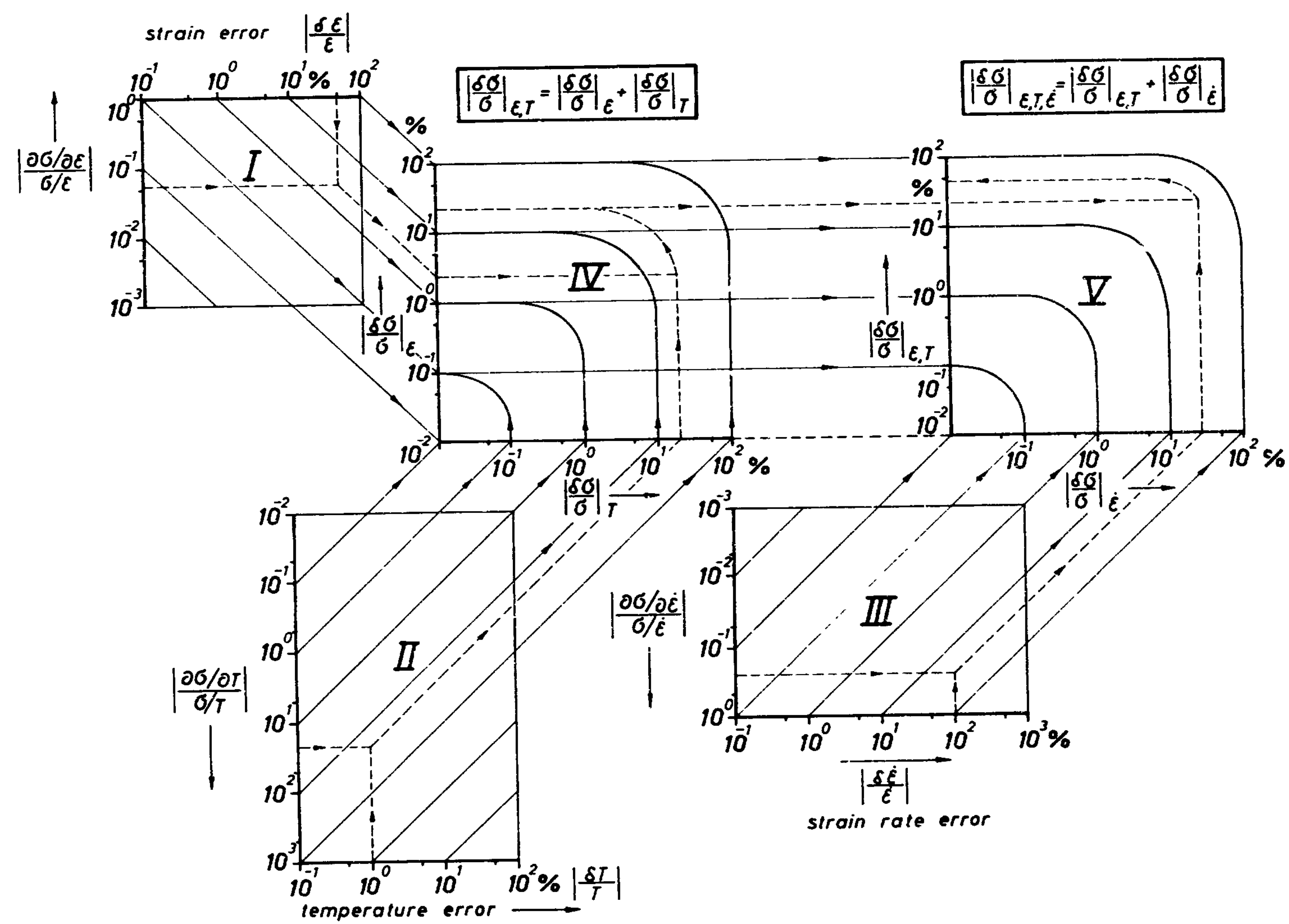

Fig. 1 Influence of the individual measurement error in strain, temperature and strain rate on the stress error

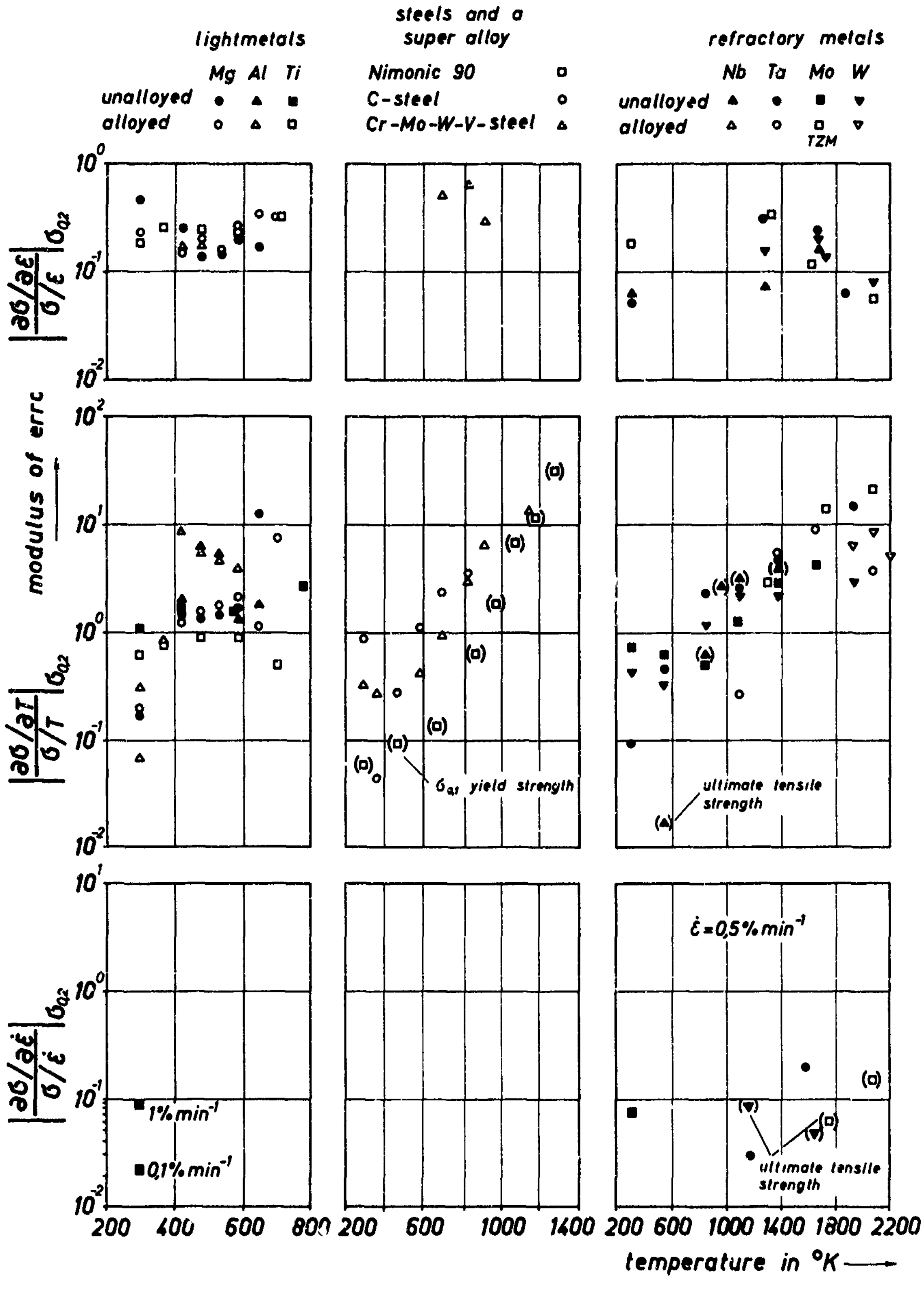

Fig.2 Influence of temperature on the moduli of error for the 0.2% yield strength

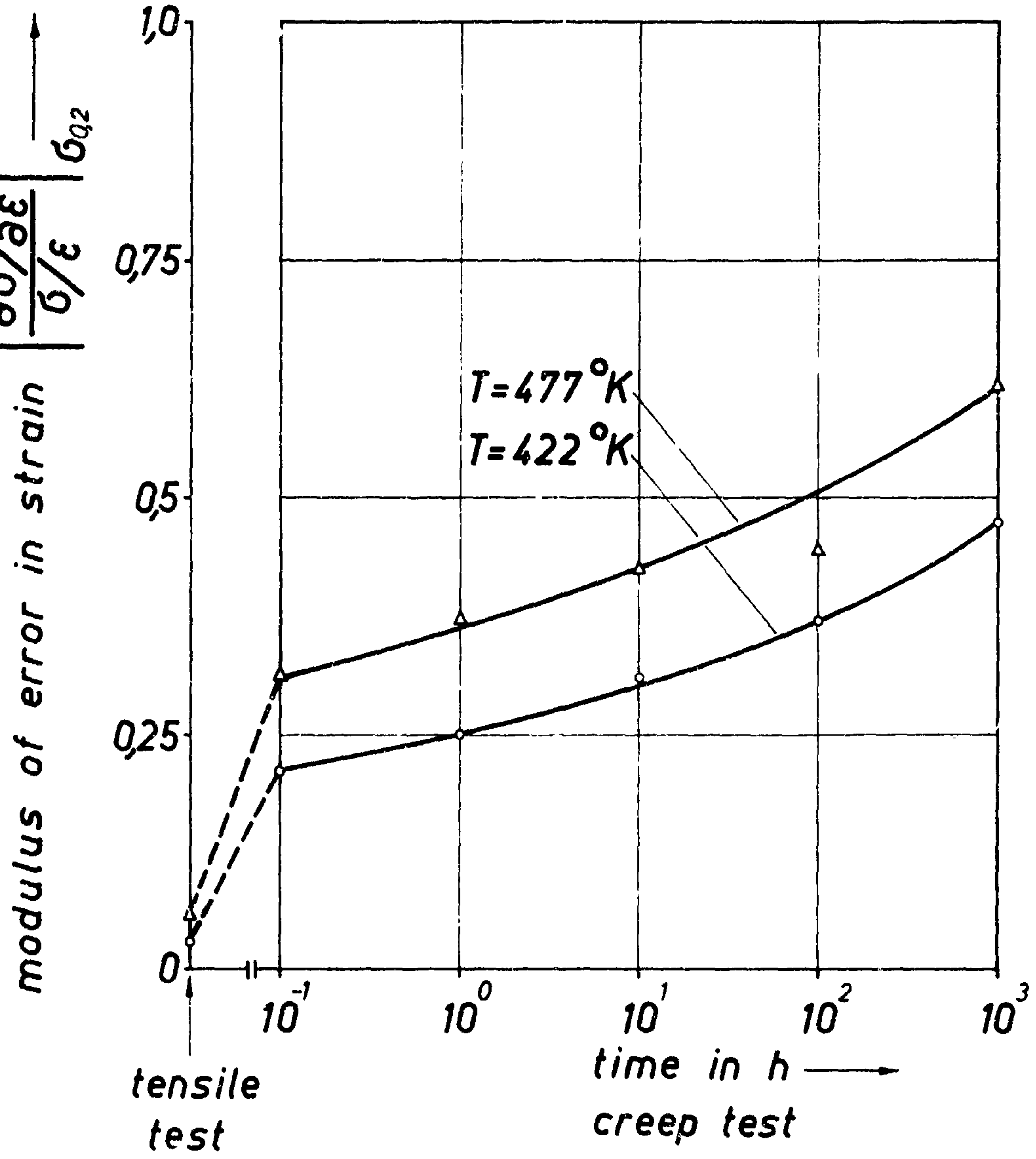

Fig.3 Modulus of error in strain in the 0.2% yield strength of an aluminum-alloy (1% Mg; 0.6% Si; 0.25% Cu; 0.25% Cr)